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ABSTRACTS

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ABBREVIATED TITLES USED IN THE ABSTRACTS

Abbreviated Title.	*Journal.*
Aircraft Eng.	Aircraft Engineering.
Ann. des Comb. Liquides	Annales de l'office National des Combustibles Liquides.
Ann. Min. Roumanie	Annales des Mines de Roumanie.
Aut. Ind.	Automotive Industries.
Aviation	Aviation.
Azerbaijan Oil Ind.	Azerbaijan Oil Industry.
Ber	Berichte der Deutschen chemischen Gesellschaft.
Brennstoff-Chem.	Brennstoff-Chemie.
Bull. Amer. Assoc. Petr. Geol.	Bulletin of the American Association of Petroleum Geologists.
Bull. U.S. Geol. Surv.	Bulletin of the United States Geological Survey.
Bur. Stand. J. Res.	U.S. Bureau of Standards Journal of Research.
Bur. Mines Bull.	U.S. Bureau of Mines Bulletin.
Chem. Age	Chemical Age.
Chem. Markets	Chemical Markets.
Chem. Met. Eng.	Chemical and Metallurgical Engineering.
Chem. Rev.	Chemical Reviews.
Chem. Trade J.	Chemical Trade Journal.
Chem.-Ztg.	Chemiker Zeitung.
Chemist-Analyst	Chemist-Analyst.
Commer. Stand. Mthly.	Commercial Standards Monthly.
Compt. Rend.	Comptes rendus hebdomadaires des Seances de l'Academie des Sciences.
Diesel Power	Diesel Power.
E. P.	English Patent.
Eng. News Record	Engineering News Record.
Engineer	Engineer.
Engineering	Engineering.
Erdöl und Teer.	Erdöl und Teer.
Finska Kem. Med.	Finska Kemistsan-fundets Meddelanden.
Fuel	Fuel in Science and Practice.
Gas World	Gas World.
Geol. Surv. Canada	Geological Survey of Canada.
Giorn. Chim. Ind. Appl.	Giornale di Chimica Industriale ed Applicata.
Ind. Chem.	Industrial Chemist.
Ind. Eng. Chem.	Industrial and Engineering Chemistry.
Inter. Petr. Tech.	International Petroleum Technology.
J.A.C.S.	Journal of the American Chemical Society.
J. Amer. Petr. Inst.	Journal of the American Petroleum Institute.
J.C.S.	Journal of the Chemical Society.
J. Chem. Soc. Japan	Journal of the Chemical Society of Japan.
J. Inst. Fuel	Journal of the Institute of Fuel.

Abbreviated Title.	*Journal.*
J. Inst. Petr. Techn.	Journal of the Institution of Petroleum Technologists.
J. Physical Chem.	Journal of Physical Chemistry.
J. Russ. Phys. Chem. Soc.	Journal of the Physical and Chemical Society of Russia.
J.S.A.E.	Journal of the Society of Automotive Engineers.
J.S.C.I.	Journal of the Society of Chemical Industry.
J. Sci. Instr.	Journal of Scientific Instruments.
L'Ingegnere	L'Ingegnere.
Mar. Eng.	Marine Engineer.
Mech. Eng.	Mechanical Engineering.
Mem. Coll. Sci. Kyoto	Memoirs of the College of Science, Kyoto Imperial University.
Mon. Petr. Roumain	Moniteur du Petrole Roumaine.
Nat. Petr. News	National Petroleum News.
Nature	Nature.
Neft. Khoz.	Neftianoe Khozianistwo.
Oil and Colour Trades J.	Oil and Colour Trades Journal.
Oil and Gas J.	Oil and Gas Journal.
Oil Bull.	Oil Bulletin.
Oil News	Oil News.
Oil Weekly	Oil Weekly.
Penna. State Coll. Bull.	Bulletin of the Pennsylvania State College.
Petr. Eng.	Petroleum Engineer.
Petr. Times	Petroleum Times.
Petr. World	Petroleum World.
Petr. Zeit.	Petroleum Zeitschrift.
Proc. K. Akad. Wetensch. Amsterdam	Koninklijke Akademie van Wetenschappen te Amsterdam.
Proc. Roy. Soc.	Proceedings of the Royal Society.
Quart. J. Geol. Soc.	Quarterly Journal of the Geological Society.
Rec. Geol. Surv. India	Records of the Geological Survey of India.
Rev. Petrolifere	La Revue Petrolifere.
Rec. Trav. Chim.	Recueil des Travaux Chimiques des Pays-Bas.
Refiner	Refiner and Natural Gasoline Manufacturer.
Roads and Road Constr.	Roads and Road Construction.
Sci. Papers Inst. Phys. Chem. Res., Tokyo	Scientific Papers of the Institute of Physical and Chemical Research, Tokyo.
Trans. Amer. Electro Chem. Soc.	Transactions of the American Electrochemical Society.
U.S.P.	United States Patent.
World Petr.	World Petroleum.
Z. anal. Chem.	Zeitschrift für analytische Chemie.
Zeit. angew. Chem.	Zeitschrift für angewandte Chemie.
Z. physikal. Chem.	Zeitschrift für physikalische Chemie, Stockiometric und Verwandschaftslehre.

ABSTRACTS.

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General.

1. PETROLEUM RESOURCES OF CHINA. *Oil News*, 1930, **28**, 534–535.

A summary is given of an article by B. P. Torgasheff in the *Chinese Nation*. In Shensi Province there are, state M. L. Fuller and F. G. Clapp, Geologists of Standard Oil of New Jersey in 1928, many permanent oil seepages. They regard Shensi and parts of Shansi, Kansu and Ordos as most favourable for development of a petroleum industry. Several wells are being operated in the Shensi Province, one producing in 1929 12 metric tons of crude daily.

At Tzeliutsing in Szechuan Province oil is found in the numerous salt wells. It has been estimated that about 100 tons of oil is raised daily with salt brine in a ratio of 1 : 4, indicating a permanent flow of petroleum from underlying strata. Petroleum is also being produced on a small scale in Kweichow. Two fields in Sinkiang were surveyed and one worked by the Russians; and oil-bearing formations have been reported in Kansu and Manchuria. Ordos seems also worthy of attention. It is concluded that serious geological exploration is warranted in at least Shensi, Szechuan and Kweichow.

E. B. E.

2. Tariff Encourages Refinery Building in Belgium. *Refiner*, 1930, **9** (10), 82.

The recently enacted Belgium tariff on crude oil and refined products is a serious inducement for the development of local refining. Whilst crude oil enters the country free, gasoline pays 70 c. per litre, and gasoline manufactured in Belgium pays 35 c. per litre only. This leaves a gross margin of about 4 c. per gal. for the local refiner, provided he could produce gasoline in Belgium on a competitive basis with the imported article. The first commercial plant in Belgium has been completed recently, and consists in a Jenkins unit which has been erected to meet with local conditions. The transport facilities are excellent, as a deep water canal, on which the plant is located, allows vessels of 27 ft. draft to dock alongside, whilst highways lead to Ghent on one side and the Belgian hinterland and Holland on the other. Moreover, there is a siding alongside. Storage tanks and loading rack for 10 cars has been provided. At present arrangements have been made for the handling of approximately 12,000 brls. of charging stock per day and distribution of 600 brls. of gasoline. Two grades of gasoline will be made, but no definite arrangement has been made for the disposal of residue. R. P.

3. European Refining To-day and To-morrow. *Refiner*, 1930, **9** (10), 68.

The relative importance of the European refining industry to the American petroleum industry is indicated by the fact that Europe has for many years taken over 50 per cent. of American refined oil exports and, aside from England, which refines about 30 million brls. per year, no real refining industry exists except in Russia. The two outstanding tendencies in European refining are increasing participation and control by governments and the shift to South America for crudes, as contrasted with previous importations of refined products from the U.S.A.

The British refining industry is an outstanding example of extensive refining facilities at a considerable distance from crude oil sources, and is explainable by the large market for finished products in Great Britain, which is the largest consumer in the world outside of the U.S.A.

The trend of mineral oil refining in Great Britain will doubtless depend in some degree on the progress made in the commercial treatment of coal tars, but more probably in the supply and price of petroleum products from other sources. In this connexion an interesting feature is the increasing quantity of refined products imported from Persia, where the Anglo-Persian Oil Company maintains a large refinery. A list of the principal refining plants in Great Britain is given, as well as the principal refineries in Rumania, Germany, Spain, Italy, Czecho-Slovakia, Belgium, France, Poland and Russia, along with a few details regarding Government control and other items of interest. Conditions in Rumania seem to be particularly bad, as there is not outlet for fuel oil, and it appears that the introduction of cracking processes would yield a considerable profit. The Rumanian Government, however, does not appear to realise the potentialities of the oil industry. Yet the cracking process has made some progress in Rumania; in fact, the Dubbs units erected by the Steaua Romana have become interesting from the fact that the longest continuous run (42½ days) by the flashing process has been completed on fuel oil cracking stock, and 115,000 brls. of fuel were cracked in this run, equivalent to 2700 brls. per day, with a total coke production of only 3 tons = 0·02 per cent. by weight, with 35 per cent. by volume of gasoline of 200° C. end point. The results of this run compare favourable with any obtained in America or elsewhere, and indicate the progress in the operating technique that is being made in Europe. R. P.

4. Italy is Building an Oil Industry based on Cracking. G. Egloff. *Refiner*, 1930, **9** (10), 75.

Under the guidance of Mussolini Italy is going farther to establish an oil industry than any other European country which does not produce crude oil within its borders. This is being done by building cracking plant and great underground reservoirs for the storage of fuel. This plan provides for the storage of heavy oils with practically no loss by evaporation, and at any time they may be converted into light oils by cracking. Conditions at present are particularly favourable to the development of these plans, as unlimited quantities of heavy fuel oils are available.

The activity of the Italians in this direction has been noticeable recently, and it is indicated that the Italian and Soviet Governments are working together to further the Italian programme, and much of the oil going to Italy is reported to be furnished by the Russian Soviet Oil Trust, "Azneft."

Whilst it is realised that great supplies of cheap oil may not always be available from Russia and Rumania, it is generally agreed that heavy oils will be available for cracking for many years to come. While the general impression of Italy's oil storage and cracking programme is of a national defence character, it is presumed that the manufacture of gasoline as a peace time industry has its advantages. The use of aeroplanes and motor-cars in Italy is increasing steadily, and the cracking process is competent to supply the necessary fuel.

The principal cracking plants in Italy are situated at Spezia and Naples, whilst another is building at Venice. These units are designed to run either on residium or non-residium system. From Rumanian fuel about 50 per cent. gasoline may be produced, and Russian residue yields over 50 per cent. by the non-residium process. Other European countries are watching the progress of the Italian programme, as cracking is of great importance to countries without natural crude supplies. R. P.

5. Oil Refining in the Soviet Union. S. S. Shipman. *Refiner*, 1930, **9** (10), 78.

Oil production and refining in the U.S.S.R. has made rapid strides during the past few years. This production is principally carried out at Baku and Grosny. At the time when the oil industry of Russia was at a very low ebb (1920) it was nationalised, and the main products were kerosine and residue, as gasoline was produced only in small quantity whilst lubricants were not produced at all. Modern plant was unknown, but since 1924–5 progress has been steady both as regards production and refining.

In addition to crude oil output, natural gas has been utilised to the extent of about 300,000 tons. Extensive introduction of modern refining methods and equipment started in 1924–25, when the demand called for an increase in the capacity of refineries. Oil exports from Russia have increased from 950,000 metric tons in 1913 to 3,626,000 metric tons in 1928–9. In pre-war exports kerosine occupied the prominent place, but in 1928–29 gasoline occupied that position. During the last three years the domestic market for Soviet oils has shown a steady increase of sales.

The construction of new plant has increased production during the past year, but in order to meet the rapidly increasing demands for gasoline reliance must be placed on cracking plant, and the construction of 14 new cracking outfits are contemplated during the current year, whilst the programme for 1930–31 calls for over 100 cracking plants, and by the end of the five years period 1932–33 it is planned to have cracking plant with a total capacity of

18 million tons, and much of the equipment is expected to come from the United States. It is proposed to bring the production of gasoline and kerosine by cracking up to 12 million tons in the last year of the five-year period. The programme calls for an annual increase of over 30 per cent.

The domestic market demands at present chiefly kerosine and residue, but it is expected that the gasoline demand will exceed the latter by the end of the five year period. To meet this demand it is proposed to construct a crude oil pipe-line from the Caspian Sea to Moscow, where 1½ million tons of heavy oil will be treated annually, whilst another refinery at Samara will treat 650,000 tons from the Emba fields. The latter plant will supply refined oil to the Volga district, the Ural district, and Siberia. Thus the majority of new refineries will produce gasoline at centres of exploitation by cracking. As a result of the improved facilities it is expected that a record increase in the production of all products will be apparent by the end of the five-year period. R. P.

6. Industrial Alcohol in Denmark. *Chem. Trade J.*, 1930, **87**, 552.

Mr. C. H. Olesen, in an article contributed to a special Danish supplement of the *Financial News* (Dec. 1, 1930), points out that the production of industrial alcohol by the Aktieselkabet de Danske Spritfabriker (The Danish Distilleries, Ltd.) has increased very rapidly of recent years, while the production of potable spirits has declined. Thus between 1910 and 1929 the production of industrial alcohol increased from 2,500,000 l. to 5,300,000 l., while the sale of potable spirit fell from 12,500,000 l. to 1,000,000 l. Potatoes are chiefly used as the raw material. The Danish Distilleries are also interested in the manufacture of potato flour and of explosives and lacquers, etc.
C. L. G.

7. Petroleum Refining in the Canary Islands. *Chem. Trade J.*, 1930, **87**, 585.

New petroleum refineries of 5000 barrels daily capacity, owned by the Compañia Española de Petroles, have just been opened at Santa Cruz de Teneriffe (Canary Islands). The refinery covers an area of 2,150,000 sq. ft., and is connected with the loading wharf by a pipe-line nearly three miles long. The plant is equipped with most modern apparatus, and can be maintained at full capacity by 150 men working three shifts of 8 hours a day. The oil requirements of the island will be supplied by this refinery, and in addition several shipping lines have indicated their intention of refuelling there. R. A. E.

8. Separation of Oil Sands by Physical Methods. W. Gründer. *Allgem. Österr. Chem.-Tech. Z.*, 1930, **48**, 105–107.

The grains of an oil sand are completely surrounded by an oil film. This film should be broken and part of the surface of the particle of sand exposed to the polar or active group of an electrolyte which has a strong wetting action upon the quartz, thereby forcing the oil from the grain. The free oil can either be floated or collected on a third oil-absorbing material. In order to break the film, the oil-sand grains may be stirred vigorously and caused to bound against plates, the friction of the quartz particles on one another assisting the mechanical process.

Oil sands from Wietze-Steinförde were extracted with carbon tetrachloride which removed 15·43 per cent. of the oil and left, on one sample, 2·55 per cent. of a solid bitumen, a black asphaltic substance. The heavy oil had a specific gravity of 0·935 and contained about 1·4 per cent. of constituents volatile at 93–110°. The following electrolytes were tested as to their ability to

drive the film of oil from the sand particles: caustic soda solution, sodium carbonate, sodium xanthate, sodium palmitate, magnesium chloride, lime water, sulphuric acid and distilled water. Only caustic soda and sodium carbonate were effective. The percentage of removal was better with the temperature of the electrolyte solution at 100° than at any lower temperature. Experiments with the oil sands in a 50 gm. flotation machine, with and without saponin, indicated only that slower stirring reduced the tendency to form an emulsion. A larger quantity of sand, 250 gms., was subjected to the action of caustic soda solution of *p*H 11·4 in a decanting jar provided with a stirrer and heated on a hot plate. By this means 95 per cent. of the oil was extracted; the total loss was 3·75 per cent. A laboratory Janney flotation machine was used for treating a kgm. sample of sand. The sand residue was free of oil, containing only the original 2·5 per cent. of solid bitumen.

W. S. C.

5. Generation of Power from Wood Waste. O. Wans, *Chem. Age*, 1930, **23**, 546–547.

The direct use of wood waste as a fuel for steam boilers is attended by the difficulty of obtaining uniform combustion, by the considerable labour involved and by the low overall thermal efficiency of such plants. (Taking the efficiency of a boiler using this fuel as 60 per cent. and a steam engine efficiency of 12 per cent., the net thermal efficiency is only 7 per cent.).

Charcoal prepared from wood can be used in a gas generator, but the process of converting wood to charcoal involves additional heat losses, labour and cost.

Modern wood waste generators embody the above dual functions, the charcoal being made from the wood and gas produced from the charcoal, thus obviating to a large degree the losses involved in the two separate processes. The overall efficiency of such a plant is 21 per cent. A plant of this type was described by the author (Inst. Chem. Eng., "Utilisation of Trade Wastes," Dec. 5, 1930). The calorific value of gas produced from wood waste in this manner is given as 155 B.Th.U.'s per cub. ft., the gas having the following composition:—

CO_2	8·5%	CH_4	3·5%
CO	26·0%	N_2	50·5%
H_2	11·5%		

The gas can be used in any gas engine of good design. Although its calorific value is lower than that of coal gas it contains less hydrogen, thus permitting of a higher compression pressure without fear of preignition. With average sawmill waste having 20 to 25 per cent. of moisture, the consumption can be taken as about 2·25 lb. per b.h.p. The gas can be used for any heating purposes, such as for steam raising, soft metal heating, heating of ovens, galvanising baths, etc. In a well-designed furnace a temperature of 1000° C. can be reached.

Generating costs given for a moderate sized producer, providing 14,000 cu. ft. of gas of 164 B.Th.U.'s (gross) per cu. ft. per hr. (equivalent to 23 therms/hr.) show a cost of 2¼d. per therm as compared with 5d. and 8d. for town gas.

C. L. G.

9A. Oil Developments in 1930. A. E. Dunstan. *J.S.C.I.*, 1930, **49**, 1048–1049.

Developments in the compression-ignition type of internal-combustion engine using Diesel Fuel indicate a possibility of serious competition to gasoline engines. Experiments are being made with aircraft as well as land engines. Diesel-engined passenger vehicles are already running on regular services.

Hydrogenation has received much attention in the petroleum industry, and technically the successful treatment of petroleum products by means of hydrogen is an established fact. Economically, however, the position is different.

The steady rise in compression ratio of the gasoline-engine and employment of higher speeds have raised the standard of fuels used, and to cope with this demand two successful attempts have been made: *firstly*, cracking has nearly doubled the normal yield of gasoline and enhanced the anti-knock value; and, *secondly*, the extraordinary effect of minute amounts of "dope," the one in wide use to-day being lead tetraethyl marketed by the Ethyl Gasoline Corporation of America.

Interest is being taken in utilisation of unsaturated hydrocarbons in the gases produced in cracking operations, such products as alcohols, glycols, ethers and other solvents already being produced on a relatively large scale.

W. A. W.

10. Creosote for Gas Making. *Chem. Trade J.*, 1930, **87**, 554.

Oil used for gas making is at present almost exclusively of petroleum origin, but it was hoped that creosote might be used for this purpose, thus providing a welcome outlet for surplus stocks. Work carried out by the Joint Research Committee of the Institute of Gas Engineers and the National Gas Council has, however, established that both light and heavy creosote oils are considerably less satisfactory than petroleum gas oils for gas making, owing to their giving a much lower yield of gas, whilst causing the deposition of large amounts of residual tar containing a high percentage of naphthalene. More hopeful results are, however, obtained from the high temperature cracking of creosote by injection into the generator. Injection of such materials into the fuel bed of a water gas generator is understood to present no technical difficulties, so that it would seem worth while persevering with these gasification experiments in view of the present surplus of creosote.

C. L. G.

11. Separation of Grit and Sulphurous Fumes from Boiler Flue Gases. J. T. Baron and J. B. Clarke. E.P. 337,436, 3.11.30. Appl., 1.7.29.

The patent describes a method of precipitating the grits and dissolving the noxious sulphurous fumes combined with the waste gases issuing from coal-fired boilers. It consists in passing the gases into a single chamber free from baffles or other obstructions presenting resistance to flow, in which the gases are sprayed with water by means of downwardly directed jets. The dimensions of the chamber are such as to reduce the velocity of the gases to 5–6 ft./second.

W. S. C.

Chemistry of Petroleum.

12. Modifications of the Interfacial Tension between Mineral Oils and Aqueous Solutions of Electrolytes produced by Certain Active Substances. E. Vellinger. *Ann. Off. Nat. Comb. Liquides*, 1930, **5**, 283–294.

The interfacial tension between mineral oils and aqueous solutions of electrolytes varies with the physiochemical properties of the two phases. Insufficient refining shows that oil substances with great surface activities are left in the oil and on ageing similar products are formed. The interfacial tension between oil and water is increased by neutral salts. The presence of organic dyes in the water phase is found to have the opposite effect and the activity is a function of the pH value.

W. S. C.

13. The Unsaponifiable Matter from the Oils of Elasmobranch Fish. Part VII., Synthesis of α-Glyceryl Ethers. G. G. Davies, I. M. Heilbron and W. M. Owens. *J.C.S.*, 1930, **131**, 2542–2546.

In Part VII. of this series an attempt has been made to determine the structures of batyl, selachyl and chimyl alcohols. It is already known that they are simple mono glyceryl ethers whose structures correspond to either (1) or (11), where R represents the octadecyl, oleyl and ceytyl radical respectively :—

$$\overset{\alpha}{\text{(1) R.O.CH}_2\text{.CH(OH).CH}_2\text{OH}} \qquad \overset{\beta}{\text{R.O.CH.(CH}_2\text{OH)}_2} \text{ (11)}$$

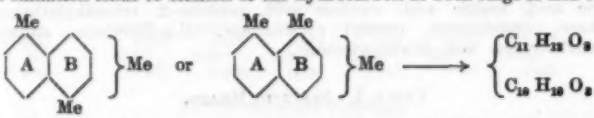
The synthesis of the α-glyceryl ethers was accomplished through the allyl ethers according to the method employed by Hilditch (*J.C.S.*, 1926, **127**, 1828). The α-cetyl glyceryl ether had m.p. 64–65° and gave no noticeable depression of melting point with a small amount of material isolated from a Japanese shark liver-oil. Subsequent work has shown, however, that the supposed pure chimyl alcohol was in reality a mixture of batyl alcohol with an unidentified substance. In this case, therefore, no direct comparison of the synthetic glyceride with the naturally occurring alcohol has been possible. Other results obtained indicated that batyl alcohol is not identical with α-octadecyl glyceryl ether and must consequently have the isomeric β-structure (11). It follows from this that, as selachyl alcohol readily yields batyl alcohol on hydrogenation, it must by analogy be the β-oleyl glyceryl ether.

W. D. S.

14. Unsaponifiable Matter from the Oils of Elasmobranch Fish. Part VIII., Structure of the Naphthalene Hydrocarbon from Squalene. I. M. Heilbron and D. G. Wilkinson. *J.C.S.*, 1930, **131**, 2546–2554.

It has already been shown in Part III. of this series (*J.C.S.*, 1926, **127**, 3136) that whereas squalene suffers complete decomposition when heated with sulphur, tetracyclosqualene yields on similar treatment a naphthalene hydrocarbon, $C_{12}H_{14}$. The oxidation of this hydrocarbon with potassium ferricyanide takes place in stages through methyl- and dimethyl-carboxylic acids to naphthalenetricarboxylic acids. The hydrocarbon is therefore a trimethylnaphthalene.

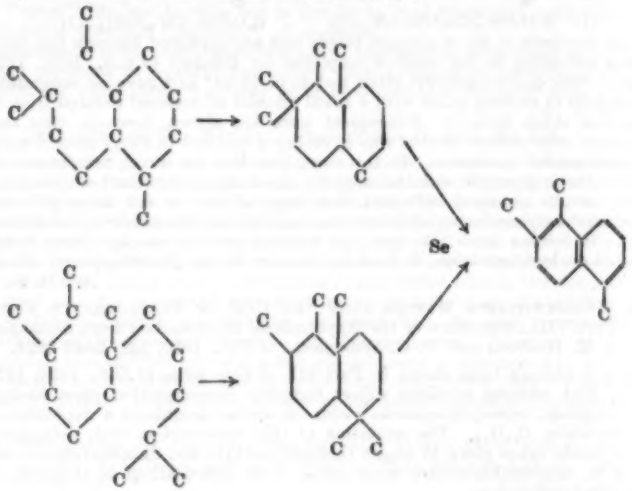
In order to obtain further insight into the position of the methyl groups, oxidation with chromic acid was employed. From the acid products a single solid acid, $C_{12}H_{12}O_2$, m.p. 126°, and containing a carbonyl group separated, and on mild oxidation with sodium hypobromite it yielded β-methyl phthalic acid. The residual oil after removal of the above acid was esterified and two solid methyl esters, m.p.'s 78–79° and 71–72°, were obtained. The higher-melting compound corresponded with an acid having the structure $C_6H_2Me_2(COMe).CO_2H$. It is thus evident that the chromic acid oxidation leads to scission of the hydrocarbon in both rings A and B :—



Among the neutral products, after incomplete oxidation, a trimethyl-β-naphthaquinone, $C_{13}H_{12}O_2$, was obtained, while with more prolonged oxidation two further products resulted. Of these, the higher-melting was identified with 1 : 6 dimethyl-α-naphthaquinone. This proves that of the two β-methyl groups present in the parent hydrocarbon, one was eliminated during the oxidation, and the third methyl group is definitely established as occupying

a β-position in the naphthalene nucleus. Therefore it follows that the hydrocarbon can only be 1 : 2 : 5– or 1 : 3 : 8 trimethylnaphthalene. The lower-melting solid accompanying the 1 : 6-dimethyl-α-naphthaquinone is probably 5 : 8– diacetoxy– 1 : 6– dimethylnaphthalene.

The 1 : 3 : 8– trimethylnaphthalene has been synthesised (*J.C.S.*, 1930, **131**, 2542), and is not identical with the hydrocarbon under review which consequently must be the 1 : 2 : 5– isomeride. The formation of the 1 : 2 : 5–trimethylnaphthalene from a sesquiterpene of the farnesene type is theoretically formulated in the manner outlined below :—



W. D. S.

15. Thermal Data on Organic Compounds. IX. A Study of the Effect of Unsaturation on the Heat Capacities, Entropies and Free Energies of Some Hydrocarbons and Other Compounds. G. S. Parks and H. M. Huffman. *J. Amer. Chem. Soc.*, 1930, **52**, 4381–4391.

Determinations of specific heat, latent heat of fusion, and, in one case only, heat of transition have been made on the following twelve compounds :—fumaric acid, maleic acid, succinic acid, pentene–2, trimethylethylene, n-pentane, cyclohexene, methyl cyclohexane, di-isobutylene, stilbene, dibenzoylethylene and dibenzoylethane.

Table I. Specific Heats.

Fumaric Acid (Crystals).

Temp. °K.	..	91·3	97·2	104·7	112·5	135·0	144·1	155·6
Cp per gm.	..	0·128	0·135	0·141	0·148	0·168	0·174	0·183
Temp. °K.	..	175·2	184·9	193·5	215·2	234·6	255·7	276·6
Cp per gm.	..	0·199	0·206	0·212	0·231	0·246	0·261	0·275
Temp. °K.	..	288·0	297·1					
Cp per gm.	..	0·285	0·292					

TABLE I.—*continued.*

MALEIC ACID (CRYSTALS).

Temp. °K.	..	91·0	97·5	103·4	124·0	136·4	149·7	163·5
Cp per gm.	..	0·124	0·130	0·134	0·150	0·160	0·170	0·180
Temp. °K.	..	177·7	189·0	199·6	220·7	247·1	275·1	281·5
Cp per gm.	..	0·190	0·196	0·207	0·222	0·241	0·262	0·268
Temp. °K.	..	287·9	294·4					
Cp per gm.	..	0·273	0·279					

SUCCINIC ACID (CRYSTALS).

Temp. °K	..	93·4	100·9	113·7	125·5	139·7	154·7	185·1
Cp per gm.	..	0·134	0·141	0·152	0·162	0·174	0·187	0·211
Temp. °K	..	215·0	244·3	260·9	265·9	272·4	274·6	276·0
Cp per gm.	..	0·237	0·262	0·277	0·280	0·344	0·295	0·289
Temp. °K	..	283·4	289·8					
Cp per gm.	..	0·296	0·303					

PENTENE-2 (LIQUID).

Temp. °K	..	136·1	152·6	169·0	201·2	230·8	260·5	275·1
Cp per gm.	..	0·436	0·439	0·443	0·458	0·474	0·492	0·504
Temp. °K	..	289·1						
Cp per gm.	..	0·518						

TRIMETHYLETHYLENE (CRYSTALS).

Temp. °K	..	92·7	93·9	97·3	101·0	104·8	107·5	111·3
Cp per gm.	..	0·238	0·240	0·247	0·254	0·261	0·266	0·277

(LIQUID.)

Temp. °K	..	143·9	152·8	173·4	183·6	201·4	203·4	213·7
Cp per gm.	..	0·448	0·448	0·449	0·452	0·459	0·461	0·466
Temp. °K	..	231·5	233·5	253·5	263·4	275·4	283·4	289·0
Cp per gm.	..	0·471	0·473	0·485	0·491	0·498	0·505	0·509

n-PENTANE (CRYSTALS).

Temp. °K	..	92·6	97·9	102·7	018·6	114·6	121·0	126·7
Cp per gm.	..	0·233	0·243	0·250	0·261	0·271	0·282	0·292
Temp. °K	..	132·2	Liquid	149·9	154·1	162·4	169·9	185·5
Cp per gm.	..	0·306	Liquid	0·465	0·465	0·468	0·466	0·473

n-PENTANE (LIQUID).

Temp. °K	..	200·4	214·9	231·2	244·5	260·2	274·8	279·2
Cp per gm.	..	0·479	0·485	0·494	0·501	0·514	0·528	0·531
Temp. °K	..	284·7	290·0					
Cp per gm.	..	0·536	0·540					

CYCLOHEXENE (CRYSTALS I).

Temp. °K	..	92·0	99·2	105·7	112·6	119·3	125·7	131·8
Cp per gm.	..	0·155	0·164	0·171	0·178	0·185	0·192	0·199

(CRYSTALS II.)

Temp. °K	..	146·5	151·2	157·1	162·3
Cp per gm.	..	0·280	0·286	0·294	0·312

(LIQUID.)

Temp. °K	..	175·2	177·1	185·4	197·1	211·9	227·0	
Cp per gm.	..	0·336	0·337	0·342	0·349	0·358	0·368	
Temp. °K	..	243·6	256·7	275·6	280·6	286·9	293·2	
Cp per gm.	..	0·382	0·390	0·407	0·411	0·419	0·425	

METHLCYCLOHEXANE (CRYSTALS)

Temp. °K	..	93·2	99·2	105·5	111·6	112·2	118·2	130·0
Cp per gm.	..	0·154	0·162	0·170	0·178	0·179	0·186	0·205

(LIQUID.)

Temp. °K	..	151·4	157·1	170·3	182·6	199·4	214·3	
Cp per gm.	..	0·339	0·343	0·351	0·359	0·368	0·378	
Temp. °K	..	229·4	244·9	260·0	275·4	285·2	294·2	
Cp per gm.	..	0·389	0·400	0·412	0·426	0·436	0·443	

TABLE I.—*continued.*

DI-ISOBUTYLENE (CRYSTALS).

Temp. °K	92·1	94·5	97·0	103·2	110·0	124·2	125·5
Cp per gm.	0·188	0·195	0·202	0·217	0·226	0·247	0·249
Temp. °K	131·3	Liquid	183·0	189·1	210·5	230·1	251·8
Cp per gm.	0·260	Liquid	0·407	0·410	0·425	0·439	0·454
Temp. °K	275·2	281·2	296·0				
Cp per gm.	0·475	0·481	0·497				

STILBENE (CRYSTALS).

Temp. °K	92·3	93·9	101·7	109·8	124·3	134·0	148·2
Cp per gm.	0·116	0·118	0·123	0·129	0·140	0·148	0·159
Temp. °K	164·9	179·6	198·8	211·7	227·6	254·4	264·6
Cp per gm.	0·174	0·188	0·206	0·219	0·235	0·262	0·273
Temp. °K	276·4	292·6					
Cp per gm.	0·284	0·301					

DIBENZOYLETHYLENE (CRYSTALS).

Temp. °K	88·6	97·5	107·6	127·0	138·6	151·5	165·0
Cp per gm.	0·111	0·117	0·126	0·141	0·150	0·161	0·172
Temp. °K	178·5	192·5	206·3	230·3	251·8	258·5	277·6
Cp per gm.	0·183	0·196	0·208	0·231	0·251	0·257	0·277
Temp. °K	284·0	291·9					
Cp per gm.	0·284	0·290					

DIBENZOYLETHANE (CRYSTALS).

Temp. °K	93·2	99·8	107·8	117·1	124·0	134·1	145·8
Cp per gm.	0·115	0·121	0·128	0·135	0·141	0·151	0·161
Temp. °K	156·0	164·6	169·5	172·7	180·3	182·4	186·2
Cp per gm.	0·171	0·179	0·183	0·188	0·203	0·207	0·219
Temp. °K	190·6	194·4	214·0	243·8	255·4	276·0	276·3
Cp per gm.	0·200	0·200	0·215	0·241	0·252	0·272	0·272
Temp. °K	281·9	289·0	296.0				
Cp per gm.	0·277	0·294	0·292				

TABLE II.

FUSION DATA.

Substance.	M.P. °K	Heat of Fusion (Cal./gm.).
Trimethylethylene ..	138·9 ..	25·74
n-Pentane	143·4 ..	27·75
Cyclohexane	169·0 ..	9·57
Methylcyclohexane ..	146·2 ..	16·24
Di-isobutylene	172·0 ..	16·83

TABLE III.

Substance.	Transition temp. °K	Heat of Transition (Cal./gm.).
Cyclohexene	186·7 ..	11·86

TABLE IV.

ENTROPIES OF THE SUBSTANCES PER MOLE.

Substance.	0-90°K	Above 90°K	Fusion.	Liquid.	S298
Fumaric Acid	12·31	27·34			39·7
Maleic Acid	12·10	25·97			38·1
Succinic Acid	13·17	28·86			42·0
Trimethylethylene	13·12	8·37	12·99	25·00	59·5
n-Pentane	13·53	9·09	13·95	25·42	62·0
Cyclohexene	11·76	18·13	4·65	17·28	51·8
Methylcyclohexane	13·08	8·77	10·90	26·59	59·3
Di-isobutylene	15·24	18·03	10·96	27·12	71·4
Stilbene (solid)	19·81	40·16	15·7		75·7
Dibenzoylethylene	24·86	51·42			76·3
Dibenzoylethane	24·79	52·81			77·6

TABLE V.

Entropy of saturated compd.		Entropy of unsaturated compd.		Entropy decrease with unsaturation, E.U.
Succinic acid	42·0	Fumaric acid	39·7	2·3
,,	42·0	Maleic acid	38·1	3·9
Dibenzoylethane	77·6	Dibenzoylethylene	76·3	1·3
2-Methylbutane	60·8	Trimethylethylene	59·5	1·3
2, 2, 4, Trimethylpentane	75·2	Di-isobutylene	71·4	3·8
Dibenzyl	64·6	Stilbene	60·0	4·6
Cyclohexane	49·2	Benzene	41·9	2·4x3
,,	49·2	Cyclohexene	51·6	—2·6
Methylcyclohexane	59·3	Toluene	52·4	2·3x2

TABLE VI.

Substance.	Heat of combustion at constant pressure.	Δ H298 Cal.	Δ S298 E.U.	Δ F298 Cal.
Fumaric Acid (S)	319,900	—193,800	—122·7	—157,200
Maleic acid (S)	326,000	—187,700	—124·3	—150,600
Succinic acid (S)	356,900	—225,100	—150·0	—180,400
Trimethylethylene	795,700	—17,300	—95·0	+11,000
n-Pentane	833,100	—48,300	—122·1	—11,900
Cyclohexene	891,700	—15,600	—104·0	—15,400
Methylcyclohexane	1,091,400	—46,800	—157·0	0·000
Di-isobutylene	1,252,000	—48,800	—175·8	+3,600
Stilbene (S)	1,764,600	+34,800	—135·8	+75,300
Dibenzoylethylene (S)	1,885,300	—33,000	—171·1	+18,000
Dibenzoylethane (S)	1,920,700	—65,900	—199·4	—6,500

TABLE VII.

FREE ENERGY CHANGES ACCOMPANYING SOME DEHYDROGENATION REACTIONS

Saturated Compound.	Unsaturated Compound.	Δ F298 Cal.
Succinic acid	H_2 + Maleic acid	+29,800
,,	H_2 + Fumaric acid	22,200
Dibenzoylethane	H_2 + Dibenzoylethylene	24,500
Ethane	H_2 + Ethylene	22,600
2-Methylbutane	H_2 + Trimethylethylene	17,600
2, 2, 4-Trimethylpentane	H_2 + Di-isobutylene	9,200
Dibenzyl	H_2 + Stilbene	15,200
Cyclohexane	H_2 + Cyclohexene	11,700
,,	$3H_2$ + Benzene	8,000x3
Methylcyclohexane	$3H_2$ + Toluene	8,000x3

NOTE.—The 15° calorie is used throughout in the calculation of these data. The free energies given in Table VI. were calculated by means of the fundamental thermodynamic equation :—

$$\Delta F = \Delta H - T \Delta S$$ A. J. G.

16. THE CONDENSATION OF HYDROCARBONS BY ELECTRICAL DISCHARGE : COMPARISON WITH CONDENSATION BY ALPHA RAYS. S. C. Lind and G. Glockler. *J. Amer. Chem. Soc.*, 1930, **52**, 4450–4461.

The condensation of methane, propane, butane and ethylene in an ozonizer under 18,000 volts potential has been studied. Measurements of the pressure changes during the reaction and analyses of the gases produced have been made and, from these, the empirical composition of the liquid condensate has been calculated. The results are summarised below in tabular form. The data for alpha rays are included for comparison :—

TABLE I.

I.—PRESSURE CHANGES.

	CH_4	C_2H_2	C_3H_8	C_4H_{10}	C_2H_4
Final Pressure (Ozonizer) / Initial pressure	1·00	0·99	1·05	1·07	0·29
Final Volume (Ozonizer) / Vol. Hydrocarbon	1·00	0·99	1·06	1·07	0·27
Max. Pressure (Ozonizer) / Initial Pressure	1·03	1·07	1·14	1·16	—
Final Volume / Vol. Hydrocarbon	1·01	1·09	1·14	1·13	0·32

TABLE I.—*continued.*

2.—FREE HYDROGEN.

$\Delta\left(\frac{\Delta \text{ Hydrocarbon}}{\Delta H_2}\right)$ (Ozoniser)	1·17	1·27	1·44	1·50	4·70
$\Delta\left(\frac{\Delta \text{ Hydrocarbon}}{\Delta H_2}\right)$ (Alpha rays)	1·32	1·31	1·54	1·44	4·18

3.—HYDROGEN-METHANE RATIO.

$\frac{\Delta H_2}{\Delta (H_2—CH_4)}$ (Ozoniser)	—	0·77	0·80	0·80	0·86
$\frac{\Delta H_2}{\Delta (H_2—CH_4)}$ (Alpha rays)	—	0·83	0·79	0·84	0·90

LIQUID CONVERSION.

	CH_4	C_2H_2	C_2H_4	C_2H_6	C_6H_6
Per cent. (Ozoniser)	54	77	74	77	93
Per cent. (Alpha rays)	30	56	82	71	90

LIQUID COMPOSITION.

Empirical Formula (Ozoniser)	CnH_2n	CnH_2n	CnH_2n	CnH_2n	$CnH_{1\cdot7}n$
Empirical Formula (Alpha rays)	CnH_2n	$CnH_{1\cdot6}n$	$CnH_{1\cdot8}n$	$CnH_{1\cdot6}n$	$CnH_{1\cdot7}n$

HYDROCARBON REACTED.

Per cent. (Ozoniser)	73	88	93	95	100
Per cent. (Alpha rays)	18	27	45	68	92

The authors emphasise the fact that, in both types of condensation, the pressure first increases to a maximum and then falls away rapidly, and they suggest that this is due to the initial formation of unsaturated compounds which are then further condensed. They conclude that "for all of the hydrocarbons studied the condensation by alpha rays and in electrical discharge have many points in common which lead to the belief that they are both brought about by the same agency." A. J. G.

17. DETECTION AND DETERMINATION OF PYRIDINE. S. B. Tallantyre. *J. S. C. I.*, 1930, **49**, 466T–468T.

The importance of proper alkalinity conditions for distillation of pyridine and "pyridine bases" is emphasised. The addition of sodium hydroxide solution before distillation until the solution is faintly pink (using phenolphthalein) is recommended.

Ladd's method of titration (*J. Ind. End. Chem.*, 1919, **11**, 552) using methyl orange and phenolphthalein appears to be the best.

The extraction of the bases from aqueous alkaline or neutral solutions (*e.g.*, ammoniacal liquors from gas works) is very complete with benzene, if salt is added until the solution is nearly saturated. The reaction of pyridine bases with the usual alkaloid precipitating agents is given with details of application and some references. W. A. W.

18. PYROLYSIS OF METHANE. R. V. Wheeler and W. L. Wood. *Fuel*, 1930, **9**, 567–574.

Reference is made to a previous paper (*Fuel*, 1928, **7**, 535; *J. Inst. Petr. Techn.*, 1929, **15**, 2A), and also Nash and Stanley (*J. S. C. I.*, 1929. 481.T.); Vysoky (*Paliva a Topeni*, 1929, p. 53); and Holliday and Exell (*J.C.S.*, 1929, **130**, 1066; *J. Inst. Petr. Techn.*, 1929, **15**, 184A).

1. "Static." Methane was heated in a 60 ml. quartz bulb at different temperatures, the pressure changes being followed by manometer readings. Equilibrium was reached in about 6 hours.

These experiments were repeated with the bulb packed with (*a*) broken quartz, (*b*) iron wire. The initial decomposition rate was increased by the increased surface and accelerated still more by the iron catalyst.

It is concluded that the decomposition temperature of methane is 650–675° C. (with or without a catalyst), and that the rapid initial decomposition is a surface effect which is checked by adsorption of a layer of hydrogen.

2. "Stream." Large volumes of methane were passed at various velocities, through heated tubes of silica, chrome iron and iron—the liquid and gaseous products being analysed in each case.

In the iron tube, decomposition proceeded so rapidly that the temperature had to be reduced from 1050° C (which was used for silica and chrome-iron) to 950° C.

The products of the reactions were passed through cotton-wool (removing naphthalene tar) calcium chloride, and then two condensers cooled to −70° C. (removing benzene), the final gaseous products being measured and analysed.

Silica and chrome-iron gave similar results—about 60 per cent. of the ingoing gas being decomposed, irrespective of the time of heating. Included in the reaction products were naphthalene, benzene, carbon dioxide, ethylene, carbon monoxide, hydrogen and methane with, of course, carbon. Butylene and butadiene were detected but no propylene or acetylene. Increase of temperature increased the rate at which the liquid products were formed. The yield of benzene ranged from 0·675 gr. per 22·4 l. of ingoing gas to 0·162 gr.

Under flowing conditions the proportion of methane converted to higher hydrocarbons increased as the time of heating decreased. Prolonged heating tended to produce carbon and hydrogen, while the shortest periods (*i.e.*, fastest rate of flow) gave hydrocarbon molecules containing 2 carbon atoms, but as the duration of heating increased the tendency was to produce aromatics. Silica and chrome-iron appear to have no catalytic effect.

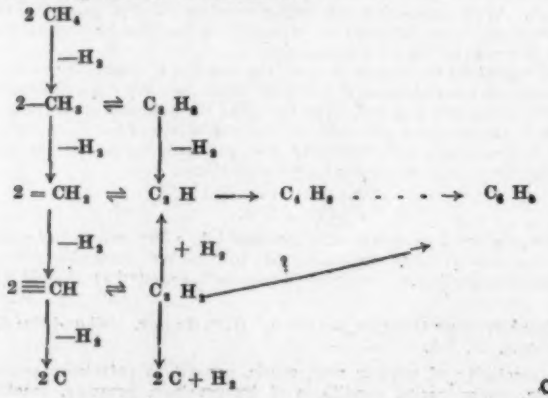
In the iron tube, no liquid products were formed—decomposition proceeding almost entirely to carbon and hydrogen.

An hypothesis is put forward for the mechanism of the decomposition of methane.

The hydrogen liberated in the initial decomposition is adsorbed on the surface, protecting it so that further decomposition must occur throughout the mass and therefore more slowly. (With iron, the protective layer is not formed.)

It is suggested that a large part of the aromatic hydrocarbons is formed from ethylene (see *J.C.S.*, 1930, **131**, 1819) and not from acetylene as suggested by Nash and Stanley.

The following scheme is advanced:—



C. C.

19. THE CONVERSION OF METHANE WITH CARBON DIOXIDE AND STEAM BY ELECTRIC DISCHARGE. K. Peters and A. Pranschke. *Brennstoff-Chem.*, 1930, **11**, 473–476.

The research described embraces the reactions of various mixtures of methane and carbon dioxide or methane and steam undergoing electric discharge at low pressures. The carbon dioxide and methane are drawn at a definite velocity from a steel flask, pumped into a discharge tube and the dry gases saturated with steam at a measured velocity at low pressures and at a temperature of 10–40°.

Various tables of results are given utilising different ratios of $CO_2 : CH_4$ at various pressures and voltages. The reactions involved showing the chemical conversion are represented by three equations, namely :—

1. $CH_4 + CO_2 = 2CO + 2H_2$ —59 Kal.
2. $CH_4 + H_2O = CO + 3H_2$ —49 „
3. $2CH_4 = C_2H_2 + 3H_2$ —91 „

With very small charges below 0·3 KW the conversion is small, and with increasing charges a pronounced increase in the conversion is always observed of the quantities of acetylene formed which approaches a characteristic maximum whilst the formation of carbon monoxide simultaneously increases. With still greater charges the acetylene concentration in the final gas falls continuously and the carbon monoxide concentration approaches a limiting value corresponding to the theoretical yields of equations 1 and 2. Curves are given showing the percentage of methane converted, and the amounts of carbon monoxide and acetylene formed in relation to the electrical charge.

With high voltages in the discharge tube and high streaming velocity, methane together with steam and carbon monoxide are almost completely converted into carbon monoxide and hydrogen. No separation of carbon occurs.

With medium voltages a large proportion of the methane is converted into acetylene.

The composition of the final gases can be varied within very broad limits by varying the initial mixture and the electrical charge. In the application of diluted methane, *e.g.*, coke-oven gas or with methane-nitrogen mixtures, a displacement of the maximum yield of acetylene with higher voltages is observed. With increasing streaming velocity of the gases an essential improvement in the expenditure of energy is established and at the same time a decrease in the total conversion.

With regard to the mechanism of the reactions, essentially more energy is expended in the formation of acetylene from methane than in the reactions shown by equations 1 and 2. The fact that the addition of steam or carbon dioxide to the methane prevents the decomposition of methane into carbon and hydrogen shows the possibility that primarily separated carbon reacts in the nascent state according to the equations :—

$$C + H_2O = CO + H_2$$
$$C + CO_2 = 2CO.$$

Also the primary decomposition of methane into a free radicle and a hydrogen atom, the conversion of carbon dioxide into carbon monoxide and oxygen, and the decomposition of water into its elements are cited as possible reactions.

W. S. C.

20. CHEMISTRY OF ORGANIC SULPHUR. H. S. Taylor. *Oil and Gas J.*, 1930, 29 (27), 38, 131.

The reactivity of sulphur compounds, present in petroleum sources and products, under varied conditions of temperature, pressure, reaction and

contact agents is but scantily known. Work in progress on Project No. 40 of the American Petroleum Institute research programme has already increased our knowledge in this direction. The application of catalysts to the treatment of sulphur compounds has been delayed by the well-known effect of sulphur compounds as poisons to catalysts being operated for the promotion of other reactions. From theoretical considerations, it is known that beneficial effects can be obtained by increasing the available catalytic surface. This should be possible by elevation of temperature and pressure. Thus it is a logical deduction that the utilisation of higher pressures of hydrogen in the hydrogenation of coal, oil and other substances, will tend to minimise the troublesome problems of sulphur poisoning. By analogy with the alcohols it would be expected that the metallic oxides would remove hydrogen sulphide from mercaptans to give unsaturated hydrocarbons and hydrogenate in the presence of metallic surfaces to give paraffins. With these theoretical considerations in view, practical investigations have been undertaken. It has been shown that metallic hydrogenation catalysts, such as reduced nickel and iron, exhibit a specific affinity for the sulphur compounds present in high-sulphur naphtha. When hydrogen was not added to the naphtha vapour, no hydrogen sulphide was evolved until "the steady state" of sulphur content of the products passed over the nickel catalyst was approached. In the initial stages the sulphur is retained by the catalyst, and at the steady state there is presumably a definite relation of sulphide to free nickel surface. With hydrogen present, the amounts of sulphur removed at each stage were increased. Iron exhibits the same type of behaviour as nickel but is less effective. In order to explain the behaviour of different naphthas, pure sulphur compounds present individually in hydrocarbon materials have been studied in contact with nickel catalysts. Mercaptans and sulphides evolve hydrogen sulphide in contact with a catalyst in the presence of naphtha vapour. Under the given conditions the mercaptans were more readily removed than the sulphides. Thiophene is only removed when hydrogen was added. The effect of other catalysts, for instance, molybdenum sulphide and cobalt oxide, is compared with nickel. W. D. S.

21. Catalytic Alkylation of Hydrocarbons, Alcohols, Organic Acids and Amines. Rheinische Kampfer Fabrik G.m.b.H. E.P. 319,205, 8.8.29.

A process is described for the catalytic alkylation of hydrocarbons, alcohols, organic acids and amines, and consists in attaching unsaturated hydrocarbons to the compounds to be alkylated with the aid of certain catalysts under the action of heat, or in causing alcohols or ethers to react with the aid of the same catalysts in the warm, on the compound to be alkylated with the simultaneous elimination of water. The terms "alkyl group" or "alkyl" are to be understood to include also the cycloalkyl and aralkyl groups, and the term "amine" to include ammonia. The catalyst employed is "activated hydro-silicate" obtained by treating bleaching earths, clays, kaolins and similar silicates with acids. Attachment of unsaturated hydrocarbons such as ethylene, propylene, cyclohexene, styrol to cyclic compounds such as benzene, naphthalene or their substitution products is ready effected by means of "activated hydro-silicates," with the formation of products alkylated at one or more nuclear carbon atoms. The following example illustrates the process.

Two parts of benzyl alcohol and four parts of benzene are heated in an autoclave to about 230° C. with one part of tonsil (activated bleaching earth) for about six hours with stirring. After separating the tonsil the reaction mixture is fractionally distilled, and the excess of benzene and the resulting

water of reaction first separated. A fraction boiling at about 260°–270° C. is next separated, which crystallises in the cold and consists of benzyl-benzene or diphenyl-methane. The latter is obtained pure with a melting-point of 26°–27° C. and a boiling-point of 264° C. by recrystallisation from petroleum ether. The last fraction consists of a mixture of isomeric di-benzyl-benzenes, which, when recrystallised from alcohol, have a melting-point of 80·5°–81·5° C. H. E. T.

22. Obtaining Aromatic Hydrocarbons. A. S. Ramage. E.P. 335,353, 3.9.29.

The process relates to the treatment of a mixture containing unsaturated hydrocarbons and a gas consisting essentially of hydrogen and carbon monoxide, for instance water-gas, with metalic iron at a temperature of from 1000° F. to 1500° F., and pressures approximating to atmospheric. Tubes of 3 ins. diameter and 10 ft. long are used in this operation, arranged in groups of three, so that the gases pass through 30 ft. of tube filled with metallic iron. With a temperature between 1100° F. and 1200° F. a suitable mixture per tube hour is 600 cu. ft. of water-gas and 400 cu. ft. of olefinic hydrocarbons of the average composition represented by C_4H_8. The iron catalyst is maintained in an active condition by the use of a suitable excess of unsaturated hydrocarbons in the gaseous mixture. The issuing gases contain large quantities of benzol, toluol, xylol, etc., which may be condensed and washed out of the gases by any of the standard methods. The stripped gas is of high heat value, usually over 1000 B.Th.U., and may be used to enrich gases of low heat value. Where any considerable quantity of paraffin hydrocarbons is present with the unsaturated hydrocarbons it is advisable to subject the gases to a preliminary treatment by passing them at a suitable temperature over ferric oxide or cupric oxide and thereby convert the paraffins into unsaturated hydrocarbons. This avoids carbon deposition due to cracking of the paraffin hydrocarbons. E. N. H.

23. Absorption of Ethylene by Means of Sulphuric Acid for the Manufacture of the Corresponding Esters, Ethers, Alcohols, etc. H. D. Elkington and N. V. de Bataafsche. Petr. Mij. E.P. 336,603 and 336,633. 4.6.29.

Ethylene is absorbed in sulphuric acid in the presence of catalysts. Osmium, iridium, platinum, ruthenium, rhodium, palladium or compounds of copper, iron, cobalt or nickel may be employed. All these substances are either brought into a soluble form in the sulphuric acid or deposited in a finely divided condition on a carrier. The compounds soluble in sulphuric acid are converted to soluble complex compounds by means of carbon monoxide or nitric oxide, while those which are insoluble are brought into solution in the sulphuric acid by means of a similar method. The absorption may be carried out in the presence of other substances which are known to accelerate the absorption, such as froth-forming substances or emulsifiers. In order to promote the solubility of the ethylene in sulphuric acid the latter may be diluted with suitable solvents, or the ethylene may be initially dissolved in a solvent. The sulphuric acid containing absorbed ethylene is subjected to treatment such as hydrolysis and distillation for the production of compounds such as alcohols, esters or ethers. W. D. S.

24. Absorption of Higher Olefines by Means of Sulphuric Acid, Phosphoric Acid or Strong Sulphonic Acids. H. D. Elkington and N. V. de Bataafsche. Petr. Mij. E.P. 336,604, 4.6.29.

In the presence of certain catalysts, olefines containing three or more carbon atoms and one or more double bonds in the molecule are much more readily absorbed by sulphuric acid, phosphoric acid or strong sulphonic acids.

Moreover, the formation of undesirable oily polymerisation products are avoided as far as possible. The catalysts used may be one or more of the compounds of the metals osmium, iridium, platinum, ruthenium, rhodium or palladium, iron, cobalt, nickel or copper. The metal must be brought into a finely divided condition on a carrier such as carbon black or silica gel. Alternatively, the metals may be converted into soluble complex compounds by passing carbon monoxide or nitric oxide through a suspension of a compound of the metal in the absorption media. The sulphuric acid or other media containing the absorbed olefines is subjected to treatment such as hydrolysis and distillation for the production of alcohols, esters or ethers.

W. D. S.

25. Treating Hydrocarbons. D. L. Thomas. U.S.P. 1,781,826, 18.11.30. Appl. 23.7.27.

A process and plant are described for desulphurising hydrocarbon oils by "atomic hydrogen," either in the cold or preferably at an elevated temperature. The most efficient temperature appears to be 400–500° C., since the velocity of reaction is then so high that only a relatively short time of contact is required. If the oils undergoing treatment suffer decomposition at their vapourising temperature, this latter may be lowered by dilution with inert gases which must not contain oxygen or steam, as these bodies inhibit the action of the atomic hydrogen.

The oil vapours are passed into a reaction chamber fitted with a refractory lining in which they come in contact with a stream of hydrogen which has passed through an electric arc maintained in the upper part of the chamber between water-cooled steel or copper electrodes furnished with highly refractory heads of tungsten or molybdenum. Current voltages of 5,000, or over are employed, and an ingenious method of striking the arc by means of a high-tension transformer is described. It is found that the necessity for adjustment of the electrodes is almost completely eliminated.

In the bottom of the reaction vessel is a pool of a fused alloy of tin and lead which serves as a desulphurising catalyst. Sulphuretted hydrogen is the main product of the decomposition of the organic sulphur compounds, and this gas can be removed by soda washing of the condensed vapours from the reaction vessel or the vapours themselves may be passed through an oxide of iron purifier.

H. B. T.

Natural Gas.

26. Conservation of Natural Gas in Relation to Some Recent Developments. S. Turner. *U.S. Bureau of Mines Information Circ.* 6392. 1930.

At present natural gas is transported over distances exceeding 500 miles, and lines are being laid to carry gas more than 900 miles from the point of occurrence. On the engineering side many mechanical devices, such as ditching machines, tractors, welding processes and improvements in pipe manufacture have contributed to this wider distribution. On the economic side the demand for steel products has been a factor of importance.

Recent developments have been associated with the Monroe-Richland Region, Louisiana, which serves the south-eastern portion of the country; the Texas and Oklahoma Panhandles, South-Western Kansas and adjacent areas, serving the Central United States; and the Californian fields, serving the Pacific Coast. From the Texas Panhandle lines are being built to serve Indianapolis, Chicago, St. Paul, Minneapolis, and intervening territory.

C

A resolution adopted by the Natural Gas Convention at New Orleans in May, 1930, recommended an intensive study of natural-gas conservation and the Bureau of Mines has carried out certain work in this connection.

Before considering natural gas as a fuel, it must be thought of as a physical agent moving crude petroleum to the surface. Between 60 and 70 per cent. of the natural gas used is produced with crude petroleum, and one of the main factors in its conservation is under control of the oil industry. Thus one industry determines the rate of production while another controls the utilisation.

Natural gas is also in demand on account of the natural gasoline contained within it. The carbon black industry also is interested in natural gas. Producers of other industrial and domestic fuels cannot, therefore, be disregarded in any consideration of natural-gas conservation or utilisation.

G. S.

27. New Helium Plant at Amarillo, Texas. C. W. Seibel. *Chem. & Met. Eng.*, 1930, **37**, 550.

Near Amarillo in Texas natural gas containing nearly 2 per cent. helium is available, and in that region the government of the U.S.A. has erected a new helium plant.

As the carbon dioxide content of the gas is rather high a preliminary treatment with a 6 per cent. solution of caustic soda is given. The gas is then cooled to about 300° F. below zero, and the helium obtained has a purity of from 40 to 80 per cent. As the gas reaches the plant from the field at a pressure of about 650 lb. further compression is unnecessary in the initial stages of helium extraction. A final stage serves to increase the purity of the helium to 98 per cent., and the gas is shipped at 1800 to 2000 lb. pressure in tank cars or cylinders.

A description of the instruments and plant is given. E. N. H.

See also Abstracts Nos. 101 and 102.

Crude Oil.

28. Apparatus for Treating Hydrocarbon Oils. J. B. Weaver, Assr. to Gyro Process Co. U.S.P. 1,763,608. 10.6.30. Appl., 15.6.30.

Crude oil is brought in contact with the vapours from a vapour-phase cracking unit whereby it is preheated and the temperature of the vapours reduced. It is then passed through a pipe still in which the temperature is raised sufficiently high to vapourize those portions that it is desired to crack, the separation of liquid and vapour taking place in an expander. The vapours, mixed with steam, are then passed through the cracking unit, the tubes of which contain ferric oxide or other catalyst which has been mixed with plastic material and worked up into bars of star-shaped cross section. In the cracking tubes the temperature of the vapours is raised to about 555°.

T. A. S.

29. Breaking Petroleum Emulsions. M. de Groote and L. T. Monson. U.S.P. 1,780,343. 4.11.30. Appl., 21.1.29.

This invention relates to the treatment of emulsions of mineral oil and water, such as petroleum emulsions, for the purpose of separating the oil from the water. The process consists in subjecting a petroleum emulsion of the water in oil type to the action of a treating agent or demulsifying agent consisting of a mixture composed of a sulphonic treating body and a wood sulphite liquor, substance or material, thereby causing the emulsion to break down

and separate into its component parts of oil and water or brine, when the emulsion is permitted to remain in a quiescent state after treatment. The various sulphonic treating bodies adapted to this process are oleo-sulphonic acid and its salts; ricinoleo-sulphonic acid and its salts; petroleum sulphonic bodies, such as petroleum sulphonic acids derived from the treatment of petroleum with sulphuric acid, and the salts of such materials; polycyclic aromatic sulphonic acids and their salts; propylated naphthalene sulphonic acid; and condensation products such as Twitchell reagents or allied bodies in which an aromatic and a fatty body or other soap-forming body are condensed and combined with a sulphonic group. H. E. T.

30. Breaking Petroleum Emulsions. M. de Groote and L. T. Monson. U.S.P. 1,780,344. 4.11.30. Appl., 21.1.29.

This process consists in subjecting a petroleum emulsion of the water-in-oil type to the action of a treating agent or demulsifying agent, thereby causing the emulsion to break down into its component parts of oil and water or brine.

The treating agent or demulsifying agent consists of a substituted aromatic sulphonic body of the type XRR_1SO_3Z, where X stands for an aromatic nucleus, and RR_1 stands for an alcohol residue or residues derived from alcohols having fewer than twelve carbon atoms in the molecule, of which at least one is an amyl residue. This reagent is also characterised by the fact that its soluble salts produce a precipitate with either soluble calcium or magnesium salts. It can be produced or obtained by introducing an amyl alcohol residue or residues, with or without other residues having fewer than twelve carbon atoms into an aromatic body. The aromatic body may consist of benzene, toluene, xylene, naphthalene, anthracene, naphthacene or similar compounds, or even halogen, nitro, hydrogenated or hydroxy derivatives of these aromatic substances. The amount of reagent required to break down an emulsion may vary from one part in five hundred to one part in twenty thousand, depending on the type of emulsion under treatment. H. E. T.

31. Breaking Petroleum Emulsions. M de Groote, L. T. Monson and A. F. Wirtel. U.S.P. 1,780,345. 4.11.30. Appl., 21.1.29.

A process is described for breaking emulsions of mineral oil and water of the water in-oil type, by subjecting the emulsion to the action of a treating agent. The treating agent employed is a substituted aromatic detergent-forming sulphonic body of the type $X.F.R.SO_3.Z$, in which X stands for a polycyclic aromatic nucleus, F stands for a detergent-forming organic residue having at least twelve carbon atoms, and R stands for an alcohol residue derived from an alcohol having fewer than twelve carbon atoms in the molecule. Detergent-forming organic bodies are of three classes and include fatty materials, such as oleic acid, or olive oil, together with resinous bodies like rosin, and also petroleum acids, such as naphthenic acids or sulphonated petroleum bodies. These detergent-forming bodies are similar, since they form soap or soap-like bodies with alkalies. The aromatic material may consist of naphthalene, anthracene, naphthacene or their halogen, nitro, hydrogenated or hydroxy derivatives. H. E. T.

32. Oil-Treating Apparatus. J. J. Fearis. U.S.P. 1,781,632. 11.11.30. Appl., 26.2.26.

The purpose of this invention is to provide an apparatus adapted for breaking water emulsions of crude oil, by application of heat with a minimum of evaporation due to the cooling surface being small compared with the heating surface.

The apparatus comprises a pair or plurality of receptacles disposed in heat exchange relation, which are designed for the reception of a substance to be treated and a heating agent, intake and discharge conductors for oil, at the upper and lower ends of the receptacles, heating agent conductors in communication with the receptacles at their upper ends, and valve-controlled exhaust conductors for said heating agents connecting with said receptacles at their lower ends, and intake and delivery conductors being provided with valves for selective use to direct the oil into and out of the receptacles, and the heating agent conductors having selective valves for the direction of the heating agent into the receptacles. H. E. T.

See also Abstracts Nos. 62 and 63.

Motor Fuels.

33. Chemists Discuss Synthesis and Refining of Gasoline. *Refiner*, 1930, **9** (10), 95.

Synthetic production of motor fuels by cracking, hydrogenation and other catalytic processes, condensation absorption and fractionation, detonation and the study of gum formation occupied the attention of the American Chemical Society (Petroleum Division) at its eighteenth annual meeting.

Much interest was evidenced in the discussion regarding the hydrogenation process by R. T. Haslam and R. P. Russell, wherein the results of working their experimental plant were given and photographs of the commercial plant now in operation at Bayway were shown.

A history of the hydrogenation of petroleum was presented, including hydrogenation of heavy residues of petroleum and coal for the production of motor fuel and other applications of hydrogenation process. It is stated that approximately 80 per cent. of the sulphur present in the raw material may be eliminated as hydrogen sulphide, and this sulphur elimination is practically always accompanied by a decrease in the gum-forming constituents in the distillates, which eliminates much of the volumetric losses incurred in treatment.

The discussion on gum formation concludes that the substances responsible for gum formation are aliphatic diolefines, cyclic-diolefines and mono- or di-olefines attached to a benzene ring. High concentration of more olefines might be involved but except in special cases gum formation is always associated with oxidation.

Stabilisation of gasoline may be effected by refinery treatment removing these classes of hydrogenations or by the use of inhibitors.

Gum inhibitors seem merely to increase the length of time before an unstable gasoline begins to break down and any complete oxidation test must involve both the determination of the induction periods and the rate of gum formation after the induction period is over. [J. Hyman and C. R. Wagner discussed some factors affecting the activity of cobalt oleate on the auto-oxidation of pentane 2. Acids and bases affect its activity.] Preliminary oxidation of the cobalt oleate produces a product which causes the disappearance of the induction period in the auto-oxidation of an olefine and an attempt was made to bring these results under a viewpoint similar to that proposed by the authors for "unpromoted" auto-oxidation. R. P.

34. Blending Natural Gasoline to Volatility Specifications. R. C. Alden and M. G. Blair. *Nat. Petr. News*, 1930, **22** (46), 107.

For practical purposes the authors have developed a very empirical expression of Raoult's law, which appears to yield satisfactory results in blending natural gasoline to vapour pressure specifications. The equation is as follows :—

$$\frac{Vn\,Gn\,Pn + Vg\,Gg\,Pg}{Gn\,Pn + Gg\,Pg} = Vb$$

where V = vapour pressure, G = A.P.I. gravity, P = volume per cent., n = naphtha, g = light gasoline and b = blend. The equation is based on the fact that the significant numbers of the A.P.I. gravities of the predominant hydrocarbons in motor fuel are roughly proportional to the significant numbers of the quotients of their specific gravities by their molecular weights. Substituting these quotients for A.P.I. gravities restores the empirical equation to the precise expression of Raoult's law. By assuming a definite relationship between the gravities and vapour pressures of the light and heavy components, it is possible to prepare a simple vapour pressure blending chart. The A.S.T.M. distillation data in the work described were determined at a barometric pressure of 745 mm. For considerably lower barometric pressures differences due to this factor must be taken into account. Charts are developed which provide a workable means of blending natural gasoline to volatility specifications under the majority of conditions. A chart is given which can be used in all cases where the finished product must meet a vapour pressure specification or in addition a specification for per cent. evaporated in A.S.T.M. distillation at 212° F. or must meet the latter specification alone. Another chart is given which can be used in practically all cases where the finished product must meet specifications for A.S.T.M. temperatures, particularly below 212° F. Two other charts are designed to facilitate the use of the last-mentioned one by expressing general relationships between several A.S.T.M. distillation points and key specifications for the components of a blend. A final one is given which may be used where the finished product must meet a specification for the 10 per cent. evaporated temperature in the A.S.T.M. distillation. W. H. T.

35. Chemical Treatment of Gasoline. M. J. Japour. *Petr. Engr.*, 1930, **2** (2), 122.

Five methods of treatment of gasoline are briefly surveyed :—

1. *Hypochlorite Treatment.*—The presence of certain forms of sulphur often makes it difficult or impracticable to use this method, also it is not always satisfactory for treating certain cracked gasolines or pressure distillates, as there is sometimes a tendency to gum formation.

2. *Vapour-phase Treatment.*—The Gray tower packed with fuller's earth gives very satisfactory results with cracked gasolines, and is very economical and efficient.

3. *Sulphuric Acid.*—It is often preferable to give a first wash with a weak acid, followed by a stronger concentration. In many cases a primary soda wash is desirable. The practice of treating the various fractions of gasoline separately is becoming more prevalent.

4. *Sodium Plumbite.*—An excessive amount of flowers of sulphur is often used. This can often be overcome by substituting rock sulphur for flowers of sulphur.

5. *Hydrogenation.*—This method will probably solve many of the treating problems which have developed in the manufacture of high anti-knock gasoline. W. L.

36. The Refining of Cracked Benzines. P. M. E. Schmitz. *Erdöl und Teer*, 1930, **6**, 510–512.

The effect of fuller's earth on hot, and soda lye (30° Bé.) on cold, crude cracked benzines is discussed, also the tendency of the benzine to darken in colour and to deposit gum.

A crude cracked benzine first lixiviated, then acid-treated, deposits only small quantities of gum, and these can be practically excluded if the crude cracked benzine is refined directly with sulphuric acid, regardless whether this treatment is followed or not by the action of floridin in the cold after neutralisation. Generally speaking, any soda lye treatment requires an after-refining with acid and consequent rectification or distillation; any fuller's earth treatment without an acid treatment requires a redistillation or rectification.

The benzine is stabilised by rectification under pressure in two stages, at pressures of 17·5 and 10 atmospheres. This rectification also removes the light hydrocarbons, *e.g.*, butane, propane, the dissolved but uncondensable gases and hydrogen sulphide and certain sulphur-containing compounds from the crude cracked benzine. The latter is then soda washed, refined with sulphuric acid of 60–66° Bé., neutralised, redistilled, and whatever refining method is applied the refined cracked benzine is again redistilled and rectified. Analyses are given of a crude cracked benzine pressure distillate containing only 70–75 per cent. of the fraction boiling to 200–205° C., in particular the Engler analysis of the ordinary crude cracked benzine, the same pressure distillate refined with sulphuric acid, the rectified pressure distillate, and a directly rectified unrefined crude cracked benzine.

Metallic oxides, fuller's earth and certain salts, like calcium chloride, etc., were utilised in the refining experiments. The results show that when the crude cracked benzine is immediately rectified a product is obtained from the cracking process which has the desired final boiling point, and for refining purposes this rectified cracked benzine is superheated directly after leaving the rectification column, and then passed over dehydrated metallic oxides, the most appropriate being red-hot iron oxide. W. S. C.

37. Recovery and Rectification of Benzols. E. Barbet. *Oil News*, 1930, **28**, 483–486, 541–543. (From a paper read to the British Section of the Institute of Civil Engineers of France.)

Figures are given for the production of benzol in gas works and in coke ovens. The production of benzol in France in 1928 was 69,000 tons, about three-quarters of which is consumed in internal-combustion engines, while in Great Britain it was about 150,000 tons, being only about 5 per cent. of the amount of imported motor spirit in either country.

The process of recovery and refining is then dealt with. A superheating-expansion method of dehydrating and distilling tars is described and then gas-stripping discussed, the bubble-tower being preferred to the scrubbing-tower. The crude benzol from either source is then submitted to a preliminary fractionation in an apparatus consisting essentially of two columns from which (1) light fractions, (2) 90 per cent. benzol, (3) toluol, (4) 120–160° C. solvent naphtha, (5) 150–190° C., and (6) a wash oil residue are obtained.

For motor benzol in France it is not even necessary to refine up to 90 per cent. benzol, but the desulphurisation is important. This, however, is accomplished sufficiently well by the primary fractionation, carbon bisulphide being separated by the process called "pasteurisation."

Washing the fractionated benzol to remove thiophene, etc., is then carried out if necessary, followed by a final rectification, which yields pure products if desired. Brief specifications are given for some of the products obtained and requirements in Germany and Belgium are mentioned. A number of plants working under these conditions exist in France, Belgium and Spain. E. B. E.

38. Method of Purifying Benzol. C. F. Hagen. U.S.P. 1,758,661. 13.5.30.

A blended distillate suitable for motor fuel is obtained by mixing discoloured commercial benzol with a heavier hydrocarbon oil and distilling the mixture until a clear distillate is obtained, rejecting the first runnings and stopping the distillation when tar oils begin to come over. T. A. S.

39. Preparing Motor Fuels. F. A. Howard, Assr. to Standard Oil Development Co. U.S.P. 1,758,898. 13.5.30.

The heavier fractions of gasoline are vapourized and mixed with a limited amount of air. The mixed gases, containing not more than four molecules of oxygen per molecule of hydrogen, are passed through a tube heated to 230–370°. The products are condensed, treated to remove gummy matter by steam distillation or dilute soda washing and remixed with the gasoline. The spirit obtained is of anti-knock value. T. A. S.

40. Treating Hydrocarbons. R. Cross, Assr. to Cross Development Corpn. U.S.P. 1,782,808. 25.11.30. Appl., 20.1.26.

The patent describes a process of refining hydrocarbon vapour or gases in which the fractionated vapourous material from the refluxing stage of a cracking system is passed through a bed of metal compound (*e.g.*, copper oxide) having an affinity for sulphur compounds. The vapours are next passed through a bed of highly adsorbent material (*e.g.*, fuller's earth) having a purifying effect but bringing about insubstantial desulphurisation relative to that effected by the metal compound. The material is finally passed through rectifying and condensing stages, any residue formed being returned to the cracking system for retreatment. W. H. T.

41. Refining Liquid Hydrocarbons. Silica Gel. Corpn. E.P. 311,285. 7.5.29.

A process is described for refining liquid hydrocarbons by bringing them into contact with a solid adsorbent to which has been added an acid (*e.g.*, H_2SO_4) which will react on the gum-forming constituents to form gummy substances. The quantity of acid is so regulated that only a portion of these constituents will react, the treatment being effected at an elevated temperature (*e.g.*, 250–300° F.) and at sufficient pressure to maintain the hydrocarbon in the liquid phase. The adsorbent is impregnated with one or more metallic oxides (*e.g.*, iron or copper oxide) and may consist of a highly-porous gel (*e.g.*, silica gel), bauxite, fuller's earth, bentonite, active carbon, adsorbent clay or an "earthy oxide." Di-methyl sulphate or anhydrous aluminium chloride may be used in place of the acid. The gums are separated from the treated materials by distillation, and the distillate again brought into contact with a solid adsorbent but at normal temperature. W. H. T.

See also Abstract No. 45.

Anti-Detonation.

42. Effect of Antiknock Materials on the Speed of Flame in a Closed Tube. Y. Nagai. *J. Chem. Soc., Japan*, 1930, **33**, 296.

Determinations have been made of the effects of Me_4Sn, Me_4Pb and Et_2Se on the velocity of flame propagation in air-hydrocarbon vapour mixtures, using a horizontal glass tube (26 mm. I.D. and 110 cm. long) closed at both ends when fired. It was found that the mean flame speeds were markedly decreased by the addition of small quantities of the above compounds and that the decrease in speed was greater than that found with open tubes, due to turbulence in the closed tubes. The addition of 0·1 per cent. of anti-knock compound reduced the speed, but a greater quantity 0·2 per cent. increased it because of speed increase in the rear part of the tube. With low initial speeds, flame propagation was smooth, giving less violent turbulence, so that the action of the compounds was weakened at the rear end of the tube where the most violent turbulence might occur. W. H. T.

43. Ignition and Combustion of Mixtures of Pentane and Air. M. Prettre, P. Dumanois and P. Laffitte. *Compt. rend.*, 1930, **191**, 414.

Benzene used as an antidetonating agent is found to raise the temperature of the first oxidation when a mixture of air and pentane are heated in a furnace, the ignition temperature remaining unchanged. $Pb Et_4$ suppresses the first oxidation completely, while amyl nitrite has no effect except to accelerate the velocity of propagation of the flame. W. H. T.

44. Water as an Antidetonant. G. W. Hobbs and M. L. Fast. *Bull. Mich. Eng. Expmt. Station*, 1930, **31**, 3–15.

Experiments were run on a standard six-cylinder engine in which water was injected through nozzles into the intake manifold to determine the effect upon detonation. The results, while not conclusive, indicate that water is a very good anti-detonant. Its effect seems to be that of lowering the temperatures throughout the cycle because of the high latent heat and high specific heat and a dilution of the fuel charge. Carbon deposits are reduced, through the prevention of chemical cracking. An increase in spark setting in direct proportion to the amount of water injected was found to be necessary. If water vapour is substituted for water the effect upon detonation appears to be lost. W. S. C.

45. Motor Fuel, Liquid Fuel. C. O. Johns, Assr. to Standard Oil Development Co. U.S.P. 1,757,837–8, 6.5.30.

In the production of anti-knock fuels the addition of a first small portion of lead tetra ethyl has more effect than the successive quantities added. Additional anti-knock effect is obtained by the addition of benzol or alcohols instead of lead tetra ethyl. Suitable mixtures are gasoline 80 per cent., benzol 20 per cent., with 1·5 c.c. per gallon of lead tetra ethyl, or gasoline 95 per cent., ethylalcohol 5 per cent., with 1·5 c.c. per gallon of lead. T. A. S.

See also Abstracts Nos. 9A, 39, 82 and 111.

Kerosine.

46. Refining Hydrocarbons. N. V. de Bataafsche Petr. Mij. E.P. 316,137, 10.7.29.

The process is for refining normally liquid hydrocarbons by means of liquid sulphur dioxide, acetone or furfural. The treatment is carried out preferably on the counter current principle and is characterised by the addition of benzol or other low boiling aromatic hydrocarbon. By this addition, the ratio of high boiling aromatic concentration in the extract layer to high boiling aromatic concentration in the raffinate layer is increased. W. H. T.

47. Treatment of Light Hydrocarbons and Kerosine. D. A. Howes, R. Scott and Imperial Chemical Industries. E.P. 336,515, 22.7.29.

The invention relates to the treatment of light hydrocarbon or kerosine obtained by the distillation of crude oil, by cracking or by destructive hydrogenation. The materials concerned are distilled in the presence of gaseous ammonia at a temperature not exceeding 300° C. in the absence of mineral acid. The gaseous ammonia is separated from the distillate and recirculated. W. H. T.

48. Treatment of Hydrocarbon Oil. Koppers Co. E.P. 336,636, 15.4.29.

The invention relates to the treatment of hydrocarbon oil to remove impurities such as unsaturated organic compounds, acid substances and the like. The previously acid-washed oil is first treated with a solution containing free alkali and alkali phenolate in such a way as to neutralise the bulk of the acid content of the oil while leaving a portion free to liberate phenols from the alkali phenolates contained in the washing solution, which phenols upon liberation dissolve in the oil. After drawing off the washing solution with the products of neutralisation, the oil is again treated with a further quantity of the washing solution sufficient to complete the neutralisation of any remaining acid substance and also phenols dissolved by the oil in the first washing stage. The second solution is withdrawn, containing the phenols as alkali phenolates but not more than very limited amounts of neutralisation products. W. H. T.

Lubricants and Wax.

49. "Oil-film," a New Artificial Lubricant. *Petr. Zeit.*, 1930, **26** (50), *Motorenbetrieb*, 12–13.

"Oil-film," produced by the S. A. Lubricating Products of Brussels, is described and recommended to displace mineral lubricating oils and consistent fats. It is prepared according to J. Woudhuysen's patented process and it contains no mineral oil, vegetable or animal constituents, differing from mineral oils in its chemical and physical properties.

"Oil-film" can be applied simply to all machines without any change occurring in the lubrication process, *e.g.*, driving and transmission of motors for airships and automobiles, steam-engines, compressors, Diesel engines, etc. It has a cooling effect on the machine and can withstand a pressure of 200 kgms./sq. cm.; moreover, it forms no vapours and prevents corrosion.

A comparison is made of its characteristic properties with a mineral oil:—

Mineral Oil.	Oil-film.
High viscosity.	Low viscosity.
Inflammable.	Non-inflammable.
Heat loss.	Small heat loss.
Unstable.	Very stable.
Change of properties after application.	Retains its lubrication properties.
Soluble.	Insoluble.
Contains acids.	Acid-free.
Contains asphalt and paraffin.	Free of asphalt and paraffin.
Mixes with benzines and other petroleum derivatives.	Immiscible with other derivatives.

Moreover, comparative experiments were carried out with castor oil, a mineral oil and "oil-film." Results show that the consumption of "oil-film" was greater than that of castor or of mineral oils, but the differences for friction co-efficient and temperature increase were distinctly less. W. S. C.

50. Transformer Oil Sludge: III. Catalytic Effects of Several Metals on the Formation of Sludge and Acids. T. Yamada. *J. Chem. Soc., Japan*, 1930, **33**, 113–146.

The relative catalytic influence of a number of metals on the formation of sludge and of acid in transformer oils has been measured by heating the oil at 140° for 100 hrs. in contact with air and with the metal. With refined oil many metals, and notably lead, zinc, iron and magnesium, tend to reduce the acidity of the oil; this, however, is due in part to the formation of metallic salts. With less highly refined oil, copper, brass, silver and lead increase the quantity of sludge formed considerably, whereas magnesium, tin, aluminium and zinc have the reverse influence. Metals which tend to accelerate sludge formation increase, in general, the formation of acid. W. S. C.

51. Transformer Oil Sludge: IV. Effects of Metallic Soaps of Naphthenic Acids on the Oxidation of Less Refined Insulating Oils. T. Yamada. *J. Chem. Soc., Japan*, 1930, **33**, 318.

Determinations have been made of the effects of metallic soaps of naphthenic acids on the oxidation of less refined switch oils. The latter were Japanese asphaltic base and American paraffin base oils, the soaps containing Mn., Fe, Cu, Ag, Co., Ni., Al., Gn., Hg., Sn., Cr., Sr., Co., Mg., K., Ba., Ce., Pb. and UO_3; 2 per cent. of the soaps was added to 5 gr. of oil and the mixtures oxidised in the open air at 120° C. for 100 hours, sludging, saponification value, viscosity and colour change being noted. Considerable sludging was brought about with Cu., Fe., Mn., Ag., Co. and Ce. soaps, while those containing Ca., Ba., Hg., K., Mg. and Sn. soaps had an anti-sludging action. The addition of 0·1 per cent. of Sn. soap is sufficient to inhibit sludge formation and colour change, but it does not reduce the break-down voltage. W. H. T.

52. Dewaxing Heavy Vacuum Distillate Stocks by the Filter-Aid Method. W. P. Gee. *Petr. Eng.*, 1930, **2** (1), 251.

With this process no difficulty is experienced in obtaining low pour test wax-cloud free lubricants from vacuum pipe still distillates. Methods of handling the filter-aid and special type filters are being developed for this process.

The following grades of paraffin-bearing oils are being commercially dewaxed by this process: Pennsylvania and Mid-Continent neutrals, vacuum paraffin distillates, vacuum overhead distillate cylinder stocks (bright stocks) all of which can be reduced to + 5 to 0° F. pour test. Bright stock which has been centrifuged can be filtered by this method to produce a finished oil of zero pour test, which is brilliant and wax cloud free at temperatures as low as –15° F. Various residual oils have been handled by this process for the past several years.

Oils are dewaxed either before or after chemical treatment. When centrifuging high viscosity distillates it is usually necessary to dewax the oil before colour treatment and experience shows that the removal of inhibitors on treating for colour frees sufficient wax to form a cloud after the oil has been stored at normal temperature. The following is a specific example of the application and advantages of the process: an overhead distillate cylinder stock was diluted with 70 parts of naphtha chilled and centrifuged in the untreated state at –45° F. giving an oil after reduction of 170 sec. viscosity at 210° F., after treating with acid and contact clay an oil of 135 viscosity at 210° F. and 20° F. pour test was obtained. This stock became cloudy under normal storing conditions. By taking the untreated centrifuged stock solution direct from the centrifuges, adding 7 to 8 lb. of filter-aid per barrel of solution and filtering through a single × chain cloth by means of a special pressure filter at –18° to –16° F., a filtering rate 2–4 gal. per sq. ft. per hour was obtained. This precentrifuged filter-aid finished product was refined in the same manner as the above centrifuged product, giving an oil of the same colour and viscosity, but of a pour test of 0° F. (A.S.T.M.). The finished stock remained perfectly bright.

Direct ammonia expansion is being installed in most modern plants for this process to permit very low operating temperatures. Generally the data given indicate that filtering at –20° F. will produce a bright oil of +5° F. pour test, and filtering at –30° F. is necessary to produce oils of zero pour test.

R. W. L. C.

53. Lubricants from California Crudes. C. O. Willson. *Oil and Gas J.*, 1930, **29** (29), 34, 35.

Lubricating oils are made in the Richfield Oil Co.'s plant at Watson, California, from Elk Hills crude, which has been found eminently suitable for this purpose after several years' research. A wax-free crude oil from selected wells is segregated. This oil has the following characteristics: Gr. 20° A.P.I., Naphtha (of boiling range 200° F. to 437° F.), 10 per cent.; Gas oil (of boiling range 400° F. to 650° F.), 40 per cent.; Lubricating distillate (gr. 18·5° A.P.I., viscosity Saybolt Universal at 100° F., 600 sec., pour point zero), 29 per cent.; Residue, steam-refined asphalt and road oil.

Special care is taken in topping the crude, and after removal of the distillates a residue of 14° A.P.I. gravity and having a viscosity of 150 sec. (Saybolt Furol) is obtained. This residue is distilled in a plant consisting of a tubular heater, a shell still and a vapour tower under vacuum, operating on a continuous system. Plenty of steam is used during distillation, and a sharp separation of asphaltic and lubricating oil materials is obtained. The products of this primary distillaton are gas oil, lubricating oil distillate and steam refined asphalt.

The lubricating oil distillate is given three acid treatments (5, 20 and 20 lb of acid per barrel), followed by a water wash and finally a caustic wash. The refined oil is redistilled on a plant similar to that described before, leaving as a residue a "cylinder stock" which is either treated with clay to yield steam refined cylinder oil, or used in the manufacture of greases, etc. The

overhead distillate is suitable as a fuel for Packard Diesel engines or for transformer oil. The other products obtained are pale and red neutral oils ranging in viscosity from 100 to 1200 sec. These neutral oils are treated with clay from Death Valley, California, and special means are employed to insure intimate contact of clay and oil. After filter pressing the oil is centrifuged before being passed to the storage tanks.

It is claimed that the methods employed yield lubricating oils which are colour stable, uniform in quality, and have exceptionally straight viscosity curves between temperatures of 100° F. and 210° F., and that the finished motor oils have cold tests below zero. The methods of treatment are sufficiently flexible to allow of variation, when necessary, to produce uniform final products. Photographs of plant and a flow chart of the operations are provided. R. A. E.

54. Modern Motor Lubrication. B. H. Lincoln and L. L. Davies. *Petr. Eng.*, 1930, **2** (1), 124.

The authors reproduce the well-known results of the more important investigations which have been carried out on thin film or boundary lubrication. Practical experience with automobiles proves that the use of "Germ" processed oils prepared by the method of Wells and Southcombe lowers the friction as much as 30 per cent. during periods of boundary lubrication. A number of automobiles have been driven relatively long distances with dry crankcases, during which time the engines have been perfectly protected by the permanent "oiliness" film established by "Germ" process oil in the previous run. R. W. L. C.

55. Work Factor Suggested as Basis for Lubricating Oil Purchases. A. E. Mockler, *Oil and Gas J.*, 1930, **29** (29), 35.

At the meeting of the Petroleum Division of the American Society of Mechanical Engineers there was a discussion of this test for the evaluation of lubricating oils. Increases in viscosity, neutralisation number, precipitation number and percentage of carbon residue of an oil when subjected to an endurance test, which simulates service conditions in a forced feed lubrication system, are expressed numerically by means of formulæ, and the average of these results is called the work factor.

The oil is subjected to test in a machine equipped with a test journal, operating at a fixed speed and bearing pressure continuously for 100 hours without means for heating or cooling the oil. The test is intended to supplement, and not replace, standard tests which, according to James G. O'Neill, United States Naval Lubricating Oil Expert, do not necessarily enable one to judge whether an oil will be satisfactory in service or not. The test is based on results obtained on lubricating oils under service conditions, and gives information on the relative stability of oils for use in turbines, Diesel engines and aviation engines. C. M. Larson, Sinclair Refining Co., said that, whilst the test is sufficient in many respects, it fails to measure the resistance to emulsification of turbine oils, and proposed the addition of demulsibility tests at 130° F. of samples taken every twenty-five hours during the test. He said that the demulsibility change would be a real measure of the stability of steam turbine oils. Another speaker described how increased efficiency and reduced costs had been obtained by standardisation of lubricants and lubricating devices. R. A. E.

56. Method of Dewaxing Oils. Oliver United Filters and J. W. Weir. E.P. 316,271. 26.7.29. Conv. (Germany), 26.7.28.

The invention relates to a method of dewaxing lubricating and other wax-bearing oils for the purpose of lowering the cold test. The removal of petrolatum and other waxes by chilling the oil and separating the wax from the chilled oil by filtration is unsatisfactory owing to the very high pressures necessary and the difficulty of maintaining a satisfactory rate of filtration. The separation of the wax from the oil is greatly facilitated by the use of a wax adherent medium of a flocculent nature, such as fuller's earth, kieselguhr, filter-cel or the like. The first step in the process consists in chilling the wax bearing diluted oil. The term "diluted oil" is understood to include any oil which remains fluid and pumpable at dewaxing temperatures of 20° F. to 40° F. The diluent may be contained in the oil in its natural state or artificially added. A finely divided wax adherent medium is then added to the chilled stock. This filter-aid material serves to separate the particles of wax and enables the treated oil to be dewaxed by passing it through a continuous filter. The wax and the filter-aid are separated by heating and treating with a suitable solvent, whereby the wax may be obtained and the filter-aid recovered in a condition fit for use in a similar cycle of operations. R. W. L. C.

57. Apparatus for Refining Used Oil. F. O. Krieger. E.P. 333,271, 10.5.29.

This invention relates to a refining apparatus for used lubricating oil, which is simple in construction and is readily transportable, and which may furthermore be installed and operated by relatively unskilled operators in garages and the like. The apparatus comprises a still, a receiver, a pipe joining the still and receiver, a chamber for condensing medium surrounding this pipe, and a device for maintaining a vacuum in the still and receiver, the vacuum maintaining device being operated by the condensing medium passing through the chamber. The apparatus also contains a clarifying tank into which passes the oil remaining after treatment in the still, and which is brought into contact with a clarifying agent in the clarifier. Diagrams illustrating the apparatus are provided. H. E. T.

58. Rectifying Lubricating Oil. M. Czarny and F. C. Frolander. E.P. 336,202. 6.6.29.

The invention relates to a method and apparatus for maintaining the quality of lubricating oil constant during the operation of an internal-combustion engine. The apparatus may be inserted in the lubricating oil circulation system of an internal-combustion engine between the oil pump and the point of supply to the moving parts. The rectifier removes the solid foreign materials by filtration and the liquid diluents by capillary separation. The liquid diluents after being removed from the lubricating oil are mixed with air and reintroduced into the engine and burned. The filtering device consists of a filter bag of bellows-like form obtained by attaching suitable fabric to alternate large and small metal rings. The evaporating device for the removal of diluents consists of a compressed wick either contained in a separate vessel or mounted centrally in the filter chamber. R. W. L. C.

59. Modification of the Physical Properties of Mineral Oils. L. Auer. E.P. 337,751. 24.4.29.

The modification of the physical properties of mineral oils is produced by the dispersion or solution of greatly modified fatty oil, fat or resin products in the mineral with the aid of heat.

The water-insoluble compounds produced by the interaction of many aromatic amines of high molecular weight, such as benzidine, *p*-phenyline diamine, and high molecular weight organic acids are useful modifying agents. Aromatic sulphuric acids, sulpho-chlorides, etc., are reagents which produce unsaponifiable products with fatty oils and resins, and such products can therefore be used as thickening agents for mineral oils for the production of lubricants. These modifying agents cause an increase in viscosity without increasing the proportion of saponifiable matter or the tendency to emulsification.

In the examples cited, the increase in viscosity of the mineral oil is produced by the addition of from 2 to 5 parts of solidified linseed oil to 100 parts of mineral oil. The solidified linseed oil is previously produced by heating it to moderately high temperatures with one of the following: lithium sulphite, 2 : 5– dichlorobenzine sulphonic acid, benzidine base, *p*-nitroacetanilide either in an open vessel or preferably in vacuo. R. W. L. C.

60. Lubricating and Other Technical Oils. I. G. Farbenind A.-G. E.P. 337,639, 28.11.29. Conv. (Germany), 29.11.28.

It is frequently desirable to enhance the viscosity of oils used for various purposes, and the materials usually employed entail several drawbacks. According to this invention these drawbacks are obviated by dissolving one or more esters, mixed esters or ether-esters of polymeric carbohydrates in the oils to be thickened. Esters, mixed esters, ethers or ether-esters of starch or cellulose which are soluble in the oil and are not sensitive to water are particularly suitable. These bodies do not tend to polymerise or resinify when strongly heated in the oil. The effect on viscosity can be seen from one of the examples given. Thus 9 parts of cellulose naphthenate dissolved in 100 parts of transformer oil raise the viscosity from 1·0° to 2·5° Engler at 100° C. The use of triethyl-cellulose is also mentioned. R. W. L. C.

61. Apparatus for Cleansing Oils. Preston Stallard and Co. and J. Malsbury. E.P. 337,982, 8.11.29.

The process claimed in E.P. 306,771 is improved by speeding up the separating operation by accelerating the cooling process. According to the invention means are provided whereby, on the cutting out of the heater, the water or like cleansing liquid can be cooled at a greater rate than the oil. One method of carrying out this process is to provide artificial means, such as a fan, associated with the lower portion of the tank in which the water settles. Normally the whole tank is surrounded by heat insulating material or lagging. H. E. T.

62. Apparatus for Fractionally Extracting Petroleum Hydrocarbons with Alcohol. T. A. Werkenthin. U.S.P. 1,781,420, 11.11.30. Appl., 30.12.27.

This invention relates to apparatus for fractionally extracting petroleum hydrocarbons with alcohol. The apparatus consists essentially of an extraction tower fitted with plates carrying contacting devices similar in construction to the conventional bubble caps. Arrangements are provided for introducing the oil to be extracted at the top of the tower and dehydrated alcohol vapour, produced during normal continuous operation by the alcohol recovery still, at a point near the base of the tower. The tower is connected with a condenser provided with a return pipe for returning alcohol to the extraction tower. Temperature control in the extraction tower is obtained by means of thermostatic devices connected with relays which operate the valves controlling the steam supply to the oil delivery pump and the water supply

to the alcohol condenser. A settling tank and a centrifugal separator are connected in series to the outlet at the base of the tower, so that complete separation of the extracted oil and the alcoholic solution may be effected. Connections and a pump are installed so that the alcoholic extract is passed to an alcohol extract still which is in operative connection with the extraction tower and separator. An alcohol dehydrator, which can be by-passed if desired, is inserted in the connection supplying alcohol from the still to the tower. R. W. L. C.

63. Fractionally Extracting Petroleum Hydrocarbons with Alcohol. T. A. Werkenthin. U.S.P. 1,781,421, 11.11.30. Appl., 30.12.27.

The process is particularly applicable to the following oils: topped crude oil, wax bearing distillates, wax bearing residual oils, heavy residual fuel oils, used lubricating oils and heavy residual oils from cracking processes. The process can be carried out in the apparatus which is the subject of U.S.P. 1,781,420 (see preceding abstract) and consists in passing the heavy petroleum oil to be extracted into the extraction vessel where it is brought in contact with superheated anhydrous alcohol vapour. The extraction vessel is designed so that the oil in a series of layers presents a large surface of contact to the alcohol vapour. The quantity of oil admitted to the extraction vessel is controlled automatically by thermostatic devices in accordance with the temperature of the alcohol vapour leaving the extraction vessel. The entire extraction process is carried out under a pressure of approximately 6 to 8 lbs. per sq. in. above atmospheric.

The complete plant for fractionally extracting petroleum with alcohol consists a number, usually four, of consecutive extraction units of substantially the same design, but somewhat differing in size, so connected that the undissolved oil which has been treated in one unit is subjected to further extraction in the next unit. The procedure and results are exemplified by the data obtained when operating on a heavy Galena Gulf Coastal crude oil of 24°Bé gravity. The light oil extracted by the first unit represents 32–33 per cent. of the crude and has a gravity of 23·8° Bé and viscosity of 207 sec. at 100° F. The oil extracted by the second unit amounts to 25 per cent. of the oil charged and has a gravity of 26° Bé and viscosity of 310–320 seconds at 100° F. The third and most important product is an oil of 600 secs. viscosity at 100° F. and of 25·2–25·4° Bé gravity and forms about 30 per cent. of the oil charged into the system. The fourth product which amounts to about 8 per cent. of the oil charged has a viscosity of 780–790 sec. and a gravity of about 23·2° Bé. Asphaltic matter remains in the undissolved residue. When paraffin bearing oils are processed, the waxy extracted lubricating oil stock can be dewaxed by cold pressing with facility as there is no formation or separation of the so-called amorphous wax on chilling. R. W. L. C.

64. Manufacture of Lubricants. F. S. Dengler. U.S.P. 1,781,444. 11.11.30. Appl. 15.10.26.

A superior lubricant may be prepared from a particular type of hydrocarbon oil by subjecting it to a carefully regulated oxidising treatment. In the illustrative example cited, a Mid-Continent crude is reduced to a residue of about 475° F. open flash point and approximately 130 sec. viscosity at 210° F. in a continuous distillation plant. The hot residue is transferred to another still fitted with air connections and maintained at 435°–450° F. or even 515° F. in some cases for 50 to 60 hours during which time air is forced through the oil in the proportion of about 30,000 cu. ft. per hour for a charge of 400 brls. of oil.

Products thus made are suitable for the lubrication of rolls and like machinery used in steel mills. Other grades range from a highly viscous liquid suitable for the lubrication of automobile transmissions to a solid material melting about 300° F. and suitable for use under most extreme conditions of high temperature lubrication. R. W. L. C.

See also Abstracts Nos. 65 and 95.

Special Products.

65. Soluble Cutting Compounds. S. J. Caplan. *Petr. Engr.*, 1930, **2** (2), 95.

Soluble cutting compounds when brought into solution with water have very high heat-absorbing properties. These solutions are non-viscous, non-rusting, harmless to the hands of operators and act as cooling agents and lubricants. The following recipes are given :—

Soluble Cutting Oil.

	lb.		lb.
Coconut oil	571	(B) W. W. rosin	271
Potash, 88 per cent.	163	E. rosin	271
Alcohol (denatured)	285	Coconut oil	232
Water	221	Pale mineral oil (100 vis. at 100° F.)	7626
		Oleic acid	361

(1) Saponify (A) without applying heat.

(2) Dissolve the rosin in 1626 lb. of mineral oil plus the oleic acid and the 232 lb. of coconut oil, with heat, and allow to cool.

(3) Mix the saponified alcoholic soap solution with the balance of mineral oil (6000 lb.).

(4) Now add the solution obtained from (2) to solution obtained in (3) and stir until perfectly clear.

Soluble Cutting Paste.

	lb.
Oleic acid	546
W. W. rosin	822
Potash, 100 per cent.	201
Coconut oil	249
Pale mineral oil (100 vis. at 100° F.)	4975
Water	3110

(1) Mix the oleic acid and rosin in a steam jacketted kettle with heat. Allow to cool.

(2) Add mineral oil and the potash dissolved in three times its weight of water. Saponify in the cold.

(3) Add coconut oil and then stir in balance of water. The finished product is creamy white.

The mineral oils and the above formulæ may be varied. For deep cuts at high speed the formulæ can be greatly improved by using mineral oil of higher viscosity. R. W. L. C.

66. Manufacture of Hydrocarbon-containing Metals and Alloys Bayerische Metallwerke A.-G. E.P. 316,103. 30.7.28.

A method of manufacture of an entirely new class of substances, namely, hydrocarbon-containing metals and alloys is described. These substances, either alone or alloyed with auxiliary metals, possess great sharpness and tensile strength, superior to all previous hard metal alloys. Metals, metal

oxides or metal alloys, preferably of the tungsten-chromium-vanadium or titanium group are heated in powdered form, in a hydrocarbon-containing atmosphere, at a temperature of the order of 1400–1600° C., but not exceeding 2500° C., for the purpose of binding the hydrocarbons with the metals or metal alloys. Ammonia gas has been found to have a highly satisfactory catalytic effect when present at a concentration of about 1–2 per cent. of the hydrocarbon atmosphere. The reaction may take place both at ordinary pressures and higher pressures. W. D. S.

67. Manufacture of Condensation Products from Olefines and Unsaturated Hydrocarbons. I. G. Farbenind. A.-G. E.P. 316,951. 6.8.29. Conv. (Germany), 4.8.28.

Olefines containing three or more carbon atoms to the molecule may be polymerised or condensed with aromatic hydrocarbons by passing over highly-porous hydrosilicates, such as Florida earth or fuller's earth, at approximately 150° C. and 15 to 20 atm. pressure. The aromatic hydrocarbon is dissolved in an inert solvent, such as decahydronaphthalene. The activity of the hydrosilicates may be increased by the addition of catalytically active halides, hydrogen halides or additive compounds of olefines and aluminium halides. The products are, in general, viscous oils similar to natural lubricating oils. A. J. G.

68. Manufacture of Condensation Products from Diolefines and Hydrocarbons. I. G. Farbenind. A.-G. E.P. 335,512. 5.7.29.

This is an extension of E.P. 315,312. Condensation is carried out using the hydrocarbon in a 3 to 4 fold excess to avoid the production of rubber-like compounds. Three examples are given: (i.) Isoprene and toluene, to give 3-methyl-5-phenyl-pentene (2) or 2-methyl-5-phenyl-pentene (2). (ii.) $\beta\delta$-dimethylbutadiene and toluene to give 2·3-dimethyl-5-phenyl-pentene (2). (iii.) Butadiene and tetrahydronaphthalene to give butenyl-tetrahydronaphthalene.

In all three cases sodium is used in the autoclave. E. N. H.

69. Manufacture of Methanol. H. Dreyfus. E.P. 335,962. 5.6.29.

This invention relates to the synthesis of methanol by subjecting mixtures of carbon monoxide and hydrogen to the action of catalysts under elevated temperatures and pressures. Catalysts which normally are sensitive to sulphur poisoning, and particularly those consisting of copper and manganese, can be successfully applied to the industrial manufacture of methanol. Methanol is prepared by passing the reaction gases, under the requisite conditions of temperature and pressure for the formation of methanol, first over one or more catalysts composed of zinc oxide or zinc oxide and chromium oxide and thence over one or more methanol catalysts sensitive to sulphur poisoning. In performing the reaction, the reaction gases may be passed successively through a number of reaction vessels arranged in series as an open system. By these means a substantially complete conversion of the reaction gases or vapours may be readily effected. An example of one method of carrying out this process is given. H. E. T.

70. Manufacture of Butyl Alcohol. H. D. Elkington and N. V. de Bataafsche Petr. Mij. E.P. 336,811. 17.2.30.

When ethyl alcohol is passed over metallic oxides at temperatures above 400° C., a yield of 20–30 per cent. of higher alcohols and their esters is obtained. The yield of butyl alcohol in these conversion products may be considerably increased if the reaction is carried out at high pressure in the presence of a

catalyst mass. This comprises a mixture of magnesium oxide and copper oxide, either in the ratio of 60 to 40 or 80 to 20. The temperature employed should be sufficient to maintain all the ethyl alcohol in the form of vapour but should not exceed 400° C. A conversion to butyl alcohol of 30 per cent. of the initial ethyl alcohol was obtained after a treatment of 2 hours at a temperature of 270° C. and a pressure of 130–140 atm. W. D. S.

71. Manufacture and Production of Ethylene. J. Y. Johnson, Assr. to I. G. Farbenind A.-G. E.P. 336,999. 24.5.29.

Ethylene is produced from acetylene by hydrogenating, using tellurium or compact nickel as catalyst. The catalyst may be used alone, or together with other catalytic or inert substances, which, in the case of tellurium, may serve as carriers, as for example, active carbon, asbestos, kieselguhr. The reaction temperature is usually between 180° C. and 500° C. and the pressure approximates to atmospheric, although the use of other pressures is covered. When a gas containing 7 per cent. of acetylene and 93 per cent. of hydrogen is passed at a temperature of 450° C. over compact metal tellurium, about 90 per cent. of the acetylene is converted into ethylene. Using nickel at 180° C. a conversion of 82 per cent. is obtained. E. N. H.

72. Polymerisation of Diolefines. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 337,019. 25.5.29.

For the production of readily soluble, plastic, tenacious products from diolefines, solvents are used and the readily soluble polymerisation products are withdrawn as they are formed. A flowing solution of the diolefine may be employed, using a solvent like petroleum ether, ether, cyclohexane or benzene. The polymerisation may be carried out at any suitable pressure. The products are often unsuitable for conversion by vulcanisation into products resembling soft rubber, but may be used for the preparation of compounds analogous to "Bakelite" or for the manufacture of threads, bands, films, etc. E. N. H.

73. Apparatus for the Manufacture of Acetylene and Hydrogen in the Electric Arc. J. Y. Johnson and I. G. Farbenind, A.-G. E.P. 337,088. 7.8.29.

Hydrocarbons in the disperse phase are passed through several electric arcs one after another. The length of the arcs are adapted in such a manner that longer arcs are used as the concentration of the hydrocarbons decreases. The acetylene can be removed at a point between any of the arcs and after the last arc. A residual gas is finally obtained which contains at most about 1 per cent. of saturated hydrocarbons. Thus the entire quantity of the hydrocarbons employed is converted into valuable unsaturated compounds and, moreover, practically pure hydrogen is obtained. The employment of hydrogen with the hydrocarbons in the disperse phase tends to suppress an undesirable formation of carbon. W. D. S.

74. Manufacture and Production of Oxidation Products of High Molecular Hydrocarbons, Waxes and the Like. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 337,130. 12.9.29.

A cooling device for regulating temperature during the oxidation of paraffin wax and similar substances consists in surrounding the reaction vessel with a pressure-tight cooling jacket into which water, or any other cooling liquid, is sprayed at such a rate that the jacket always contains some of the liquid phase. In the event of there being a sudden evolution of heat from the reaction mixture the pressure in the cooling jacket is released. The resultant rapid evaporation gives an excellent cooling effect. A. J. G.

75. Oxidation of Hydrocarbons in the Gaseous Phase. Hans Harter. E.P. 337,407. 24.6.29.

Hydrocarbons, more especially methane, may be oxidised in the gaseous phase at 1000° C. to give small amounts of formaldehyde. When the oxidising agent, directly before coming into contact with the hydrocarbon, is passed through a flaming arc, nitrogen oxides are formed. These for the most part decompose when passing out of the range of the flaming arc and are thus capable of initiating and supporting the oxidation of the hydrocarbon to formaldehyde at considerably lower temperatures, for instance, between 100° C. and 500° C. The formation of the nitrogen oxides is promoted by catalysts such as quartz, zirconia, thoria, boron nitride and so forth, whilst the formation of formaldehyde is promoted by catalysts such as copper, silver, cobalt, manganese, particularly in combination with or deposited on silica gel or active carbon.

An apparatus is described for carrying out this process. The nitrogen containing oxidising gas is passed through a number of slots and passages of an externally cooled pipe. Electrodes for the production of a flaming arc are so arranged that the electric flaming arc passes through the said pipe, preferably made of the catalytic material. The hydrocarbon gases are introduced into a chamber, in which they come into direct contact with the gases leaving the flaming arc, by means of a number of passages in a double-walled tube. W. D. S.

76. Polymerisation of Butadiene 1 : 3, Homologues and Analogues Thereof. A. Carpmael, Assr. to I. G. Farbenind. A.-G. E.P. 337,460. 2.8.29.

The polymerisation of butadiene is regulated by placing an alkali metal in a small container provided with small holes or slits, and this is then introduced into the main reaction vessel. The small vessel first fills with polymerisate, which swells in volume as the polymerisation proceeds and a mixture of alkali metal and polymerisate is extruded into the main body of the hydrocarbon. The reaction is carried out in a rotary autoclave at a temperature of 30° C. It is claimed that a highly elastic, uniform polymerisate is produced by this method. A. J. G.

77. Manufacture of Acetone. H. D. Elkington and N. V. de Bataafsche Petr. Mij. E.P. 337,566. 10.10.29.

Acetone is manufactured from isopropyl alcohol by passing the vapour of the latter over a catalytic mass at a temperature below 400° C. The catalyst comprises a mixture of substances known to have a favourable catalytic effect on the reaction (copper, manganese peroxide, barium peroxide, metals of the platinum group, zinc oxide, cadmium oxide, uranium oxide, blue tungsten oxide, etc.) with about 1 per cent. of one or more of the following substances, zinc oxide, thorium oxide, sodium carbonate, cerium oxide or zirconium oxide. These latter substances prolong the life of the catalytic mass. W. D. S.

78. Manufacture and Production of Valuable Condensation Products from Diolefines. J. Y. Johnson. E.P. 338,152. 14.5.29.

Diolefines such as butadiene, isoprene, dimethyl butadiene, etc., when treated with substantial amounts of inorganic oxygen-containing acids of sulphur or of phosphorus or their organic derivatives, *e.g.*, sulphuric acid, benzene sulphonic acid, toluene sulphonic acid, *p*-toluene sulphochloride, benzene sulphochloride, produce valuable condensation products. Oily,

resinous or solid reaction products are obtained whose consistency and hardness can vary within wide limits. Mild conditions at low temperatures in low concentration, or for short periods of time yield low molecular products varying from oily-liquid to semi-solid in consistency. Longer or more energetic action produces high molecular products, which vary in hardness. The reaction can be performed below 0° C. or under high pressures.

The products so obtained can be utilised as adhesives, as additions or as softening agents for paints and lacquers, as additions in the manufacture of rubber-like products, for the manufacture of films, foils, threads, bands and moulded articles. W. S. C.

79. Apparatus for Hydrolyzing Acid Sludge. F. L. Maker, J. L. Cooley and C. F. Tuttle, Assrs. to Standard Oil Co. U.S.P. 1,760,246. 27.5.30. Appl., 18.4.27.

Acid sludge is hydrolyzed at about 165° under a pressure of 50 lb. per sq. in. The reaction is carried on in a vessel fitted with a baffle from one side of which weak clear acid can be removed. Tar suitable for fuel is removed from the upper layer on the other side of the baffle. T. A. S.

See also Abstract No. 27.

Cracking.

80. Effect of Time and Temperature in Treating Cracked Distillate. R. B. Pierce. *Nat. Petr. News*, 1930, **22** (46), 121.

It is shown that the longer the time of agitation of acid with the cracked distillate, the greater the reduction of sulphur, the poorer the colour of the rerun gasoline and the greater the amount of acid formed at the rerun stills. The greater the quantity of steam used during distillation, the better is the colour of the distillate and the less the acid formation at the rerun stills. The amount of steam however has little effect on the sulphur content of the rerun overhead. A storage time of one day or more before rerunning improves the colour of the distillate and reduces acid formation of the rerun stills, the sulphur content not being affected. A water wash before caustic neutralisation does not affect colour or acid formation at the rerun still, but the sulphur content is slightly lowered. For caustic washing, a caustic strength higher than 2° Bé. is preferable, but any strength above 4° Bé. will give the same results with regard to sulphur, colour and acid formation. A low temperature treatment gives better colour than a high temperature treatment and the acid formation on rerunning is markedly less, but the sulphur is slightly lower on the distillate treated at a high temperature. Settling time before caustic neutralisation increases acid formation at the rerun stills, affects the colour of the distillate but lowers the sulphur content slightly. W. H. T.

81. Vapour-Phase Cracking in England. W. McKechnie Robson. *Refiner*, 1930, **9** (10), 73.

A plant has been erected in the River Humber at Killingholme under licence to operate the Gyro vapour-phase cracking process in association with the Low Temperature Carbonization Co., as it has been found that this cracking process is applicable to the oils obtained in low temperature carbonization of coal.

The Killingholme refinery is self-contained and is fully equipped for complete operation. The plant operates at atmospheric pressures and has a large factor of safety. It is simpler than many types of cracking units, obviating high pressure engineering so that maintenance costs are low. A wide range of raw material can be used without effecting the products whilst the plant may also be used as a combined topping and cracking plant. R. P.

82. Data on Low-Temperature Cracking. G. Egloff and E. F. Nelson. *Oil and Gas J.*, 1930, **29** (26), 56, 188.

The data given shows that excessive temperatures are not necessary to produce high anti-knock motor fuels from almost any kind of charging stock. Six different oils, covering a wide range of stocks, were tested. They were cracked at pressures ranging from 210 to 350 lb., and at temperatures of 925° to 967° F. They produced yields of gasoline varying from 53 to 78 per cent. of the charging stock and the gasoline produced showed a knock rating varying from 54 to 71 per cent. benzol equivalent. The knock rating tests were conducted by means of the "Ethyl series 30 motor."

The cracking process in each test was the flashing method. Tables are given showing the results of laboratory tests on each of the charging stocks, the operating conditions for the different cracking stocks and the gasoline, flashed residue and gas yields.

The flashed residues from the cracking operation are superior in quality, from a fuel oil standpoint, to the charging stocks.

The tests show that the same process and the same mode of operation, in the same cracking unit produced successful results from six different stocks, not only from the standpoint of yield, but from that of anti-knock quality as well. W. L.

83. Preventing the Formation of Coke in the Cracking of Mineral Oils. S. Seelig. E.P. 336,822. 6.11.29.

The products of cracking, directly after leaving the reaction vessel, are brought into contact with inert gases or vapours in order to prevent further reaction and the formation of carbon or coke in such products. W. H. T.

84. Cracking Hydrocarbon Oils. Marks and Clerk, for Panhandle Refining Co., Texas. E.P. 337,380. 29.6.29.

A method is described for cracking hydrocarbon oils at sub-atmospheric pressure, and details of a plant are given.

Charging stock heated in a pipe still under an absolute pressure of 15 to 60 lb. is dropped to 12–14 lb. absolute in a flash pot and the vapours are drawn through a cracking coil contained in the same furnace and heated to 1000–1200° F. under an absolute pressure of 6–14 lb.

The low pressure ensures high velocity of gas flow giving turbulence and greater heat transfer per unit area of heating surface.

The vacuum pump is so installed that the vapours may be drawn from the cracking coils and passed into the fractionating and cooling towers or the negative pressure may be applied to the gas separator at the end of the plant and the vapours therefrom passed into an absorption tower.

The vapours from the cracking coils are passed through a series of three fractionating and cooling towers, of which the second and third have bubble-cap plates. The condensate from the first tower is removed from the system, that from the second tower being pumped partly into the first column and partly through cooling coils in the top of this column and ultimately into the flash pot.

The condensate from the third tower, after cooling in a heat exchanger, is pumped partly into the absorbtion tower and partly through cooling coils in the tops of the second and third columns. The vapours in this tower are cooled by the introduction of oil from the bottom of the absorbtion tower warmed by passing through the heat exchanger, and are then passed to a condenser and separator.

The oil from the bottom of the absorber is stripped of its light fractions in the third column and it is claimed that by this method of working a more stable product is obtained than gasoline produced in the ordinary way and with the light strippings from the absorbing oil added to it afterwards.

H. B. T.

85. Process for the Regeneration of Aluminium-Chloride Sludge. E. P. Phillips and J. G. Stafford, Assrs. to Sinclair Refining Co. U.S.P. 1,760,962. 3.6.30. Appl., 13.3.23.

Aluminium sludge from cracking operations is first steamed whereby hydrochloric acid and oil are removed, and a porous mass of aluminium oxide and coke is produced. This is dried and some gas produced by heating it with air in a gas producer and the mass is finally burnt in contact with air and chlorine, whereby anhydrous aluminium chloride is produced. The chlorine is prepared from the hydrochloric acid recovered in the steaming operation.

T. A. S.

86. Useful Products from Hydrocarbons. H. R. Rowland, Assignor to C. and C. Developing Co. U.S.P. 1,779,402, 21.10.30. Appl., 21.11.23.

The production of oils of low specific gravity and boiling point from mineral oils of relatively high specific gravity and boiling point is brought about by a combination of heat treatment and the use of an electrical discharge of frequency below 10,000 cycles per second. The use of low frequencies is stressed and mention is made of commercial frequencies in the range 25 to 150 cycles per second.

In an example, use is made of a preheater maintained at 850 to 900° F., and this is in the form of multiple narrow tubes to obtain good heat transference. The vapours then pass through the reaction tubes in which fine wire electrodes are suspended axially. The temperature in the reaction tubes is between 800° and 900° F. A 65 per cent. yield of crude motor fuel is claimed.

Carbon deposition is minimised by mixing the charging stock with some hydrogen-containing gas or natural gas, and in the given example 3 to 4·5 cu. ft. of natural gas are introduced into each gallon of kerosine distillate vaporised.

Pressures near atmospheric are usually employed but the use of other pressures is covered.

E. N. H.

87. Hydrocarbon Oil Conversion. C. P. Dubbs, Assr. to Universal Oil Products Co. of Chicago. U.S.P. 1,779,465, 28.10.30. Appl., 30.7.27.

A process and plant are described for the production of a motor spirit of correct boiling range and of high anti-knock value by cracking at substantially atmospheric pressure.

The process is essentially a counter-current one in that the charging stock passes through a series of four chambers in each of which it meets a stream of cracked gases passing through the series in the opposite direction. On leaving the fourth chamber the stripped charging stock, together with the heavy fractions of the cracked gases, is pumped through a tube still into a reaction chamber at a temperature of about 1000° F. Heavy residue is

drawn off from the bottom of this vessel, and the vapours are taken backwards through the series of chambers which are maintained at temperatures of approximately 700°, 650°, 550° and 450° F.

Pumps and blowers for the transfer of charging stock and cracked gases are installed between the chambers, and means are provided whereby the cracked gases can be taken from the top of any one but the last of the chambers and passed through another tube furnace before being introduced into the next chamber of the series. H. B. T.

88. Splitting Up of Mineral Oils. M. Melamid. U.S.P. 1,779,486, 28.10.30. Appl., 12.3.25.

This patent deals with the cracking of the lower boiling fractions of crude oils and tars in the presence of hydrogen or of hydrogen containing gases, and with the utilisation of a contact material such as tin bismuth or antimony which is molten at the temperature of the reaction.

The tar is atomised by means of hydrogen-containing gases into a vessel, in the bottom of which is the liquid contact material at a temperature of 300–400° C. The heavier, unvaporised portion of the feed is continuously removed from the vessel, while the vaporised portion undergoes cracking without deposition of carbon. H. B. T.

89. Treating Heavy Hydrocarbon Oils. A. A. F. M. Seigle. U.S.P. 1,779,829, 28.10.30. Appl., 12.12.23 and 9.8.27.

This patent relates to improvements in the method of cracking hydrocarbon oils described in U.S.P. 1,702,275.

The oil vapours at a temperature of 350° C. and at atmospheric pressure are passed upwards through a vertical retort furnished with a central vertical flue down which furnace gases are led. The annular chamber of the retort is fitted with a number of trays and baffles so arranged that the oil vapours circulate around the trays which are filled with metallic chips or turnings. The metallic fragments are machine shop waste and may consist of iron, nickel, copper, etc.

The vapours in their passage through the retort are gradually heated to a temperature of 500°–700° C., and then enter a cooling and expansion apparatus. The cooling is effected by a closed system containing water boiling under pressure, and the temperature is reduced to about 210° C. The expansion chamber is likewise fitted with shelves covered with metallic fragments.

The heavy fractions condensed in this chamber are returned to the still with the charging stock, and the vapours pass to a second similar cooling and expansion chamber where the cooling medium is water boiling under pressure at a temperature of approximately 135° C.

The condensate from the second chamber is passed back to the first one and the cracked vapours are taken overhead. H. B. T.

90. Treating Hydrocarbons. E. A. Prudhomme, Assr. to Soc. Int. des Procedes Prudhomme. U.S.P. 1,780,536, 4.11.30. Appl., 5.6.26.

A continuous process for treating heavy liquid hydrocarbons is described. The material is atomised and then cracked in a heated chamber containing a catalyst purifier, the resultant products being condensed. The hydrocarbon to be atomised is heated by returning the heavy fractions to the charging stock. The coke deposited during cracking is burnt, while the residual gases are used to assist in the atomisation of the charge, the residual gases being heated by the combustion of the coke. The uncondensed products resulting from the cracking are conducted through a plurality of catalyser stages, and unretained gases are finally stored in a gasholder. W. H. T.

91. Cracking Petroleum Oil. R. T. Pollock, Assr. to Universal Oil Products Co. U.S.P. 1,781,128, 11.11.30. Appl., 9.3.27.

Improvements in cracking petroleum oils are described in which the reflux condensate can be separately cracked as part of a continuous process and under different conditions of temperature and pressure from the crude oil. High boiling-point oil is subjected to a primary cracking treatment and at least three components separated—*e.g.*, constituents having the characteristics of gasoline, intermediate constituents heavier than gasoline and residuum. The gasoline-like constituents and the residuum are removed from the cracking zone, and the intermediate constituents heavier than gasoline are cracked in successive stages; the desirable products—*i.e.*, gasoline and the undesirable components—are removed from the cracking zone as formed, and only the intermediate products are subjected to a prolonged cracking treatment. W. S. C.

92. Refining Hydrocarbons. E. C. Herthel, Assr. to Sinclair Refining Co. U.S.P. 1,781,388, 11.11.30. Appl., 29.2.28.

The invention relates to improvements in refining cracked gasoline in the vapour phase by passage of the gasoline vapours through an adsorptive catalyst such as fuller's earth. This treatment causes polymerisation of unsaturated constituents unsuitable as components of the refined spirit. The polymers produced by the vapour catalyst contact exceed 3 per cent. by liquid volume of the refined gasoline product after separation of such polymers. The improvement consists of carrying out the polymerisation without separation or discharge of such polymers from the operation. The total vapour is then discharged directly to a rectifying zone where polymers and other high boiling constituents are condensed. The remaining gasoline vapours are passed upwards counter-current to the condensed polymer mixture and finally condensed to form the refined gasoline product. W. H. T.

93. Cracking of Oils. E. C. Herthel and E. W. Isom, Assrs. to Sinclair Refining Co. U.S.P. 1,781,390, 11.11.30. Appl., 7.1.25.

This invention relates to an important method of operating circulatory pressure stills provided with reflux towers. It includes an improved method and apparatus for distilling hydrocarbon oils under pressure. A body of oil is maintained in a bulk supply tank, and is pumped upwardly through a vertically arranged tubular heater located away from the bulk and back to the supply tank. The oil is maintained at a cracking temperature throughout the cycle. The vapours from the bulk supply tank are subjected to a refluxing operation, and the reflux and admixed charging stock from the refluxing operation are forced upwardly into the oil circulating from the bulk supply tank through the heater. Connections are fitted for conducting vapours from the bulk supply tank to the reflux tower, and a pump for injecting reflux from the reflux tower upwardly into the lower end of the heater. W. S. C.

See also Abstracts Nos. 4, 28, 36, 40, 62, 63, 105 and 106.

Hydrogenation.

94. Hydrogenation of Mineral Oils. I. N. Beall. *Refiner*, 1930, **9** (10), 147.

The first broad requisite of the perfect oil conversion process is that such a process can convert any crude oil into 100 per cent. by weight of motor fuel or other desired product. This would necessarily mean no formation of coke

or fixed gas. The process would operate continually and 100 per cent. efficiency assumes the conversion of the cheapest raw material into the highest percentage of best quality product. Taking this standard of comparison, all cracking processes have failed to meet the standard because the laws of equilibrium demand that fixed gases rich in hydrogen be formed, which in turn demands a sacrifice of material rich in carbon to provide the hydrogen; hence the production of tar and coke. This effect results in discontinuity of the process. Cheap raw material, such as asphaltic crudes, give low yields of spirit, and much tar and coke, and are thus uneconomical, lowering the efficiency of the process. Hydrogenation, on the other hand, holds possibilities of more than 100 per cent. by weight of the raw oil. Hydrogenation in the broadest sense is not limited to supplying free hydrogen, but includes the addition of hydrogen in a combined form such as the unsaturated gases and partially oxidised hydrocarbons of higher hydrogen-carbon ratio than the raw oil.

With the partially oxidised hydrocarbons it is theoretically possible to build up on a crude oil stock, as it is possible to synthesise motor fuel direct from such compounds, for example, in the Synthol and Patart processes. The possibilities of hydrogenation of crude stock with a mixture of carbon monoxide and hydrogen should be infinite, and such a process would be flexible. Water-gas reactions are applicable to refinery and cracking gases as well as natural gas, and are convenient methods for the production of hydrogen. The fact that such gases contain carbon monoxide and methane does not prevent their use as hydrogenating gas for crude oil stock, although for ammonia synthesis the gas is freed from carbon monoxide. Such water-gas reactions require a catalyst, and chrome zinc oxides are much used for this purpose. The use of hydrogen prevents the formation of coke if correctly applied, and the addition of oxidised hydrocarbon would help to prevent the formation of carbon in a continuous high-pressure process for oil hydrogenation. The raw materials are low-grade crude oils, residues and other materials unsuitable for cracking; and for the production of hydrogen there are waste refinery and cracking plant gases, natural gas and steam. The initial cost for plant is necessarily high, due to the high pressures and extra plant required for the production of hydrogen and its treatment.

Different investigators have put forward processes using steam as a source of hydrogen, but synthesising pressures just begin where most of these investigators left off, and high pressure is the most powerful means so far devised for the purpose. Many claims have been made for the use of natural gas as hydrogenating material for oil, but in almost every case it is found that no benefit could be attributed to other than oil cracking or physical combination, as methane and ethane are chemically inert under ordinary cracking conditions. There are two methods of attacking this problem, either the activation of the gas, or of the oil, preliminary to conversion, and the former appears more logical because of the few and simple compounds involved. The use of steam for heat conversion includes cracking and oxidation as the reaction

$$C_nH_{2n+2}+nH_2O \rightleftarrows nCO+nH_2$$

involves dissociation of both hydrocarbon and steam with subsequent oxidation of the carbon. The reverse reaction is one of reduction or hydrogenation, such as

$$CO+3H_2 \rightleftarrows CH_3OH+H_2$$

which takes place best at a high pressure and low temperature in the presence of a catalyst.

Hydrogenation is a chemical principle capable of widespread application, as it is not one process but is the basis of many processes, and commercial

plant where pressures of 15,000 lb and temperatures of between 500 and 600° C. is available. There would appear to be certain advantages to vapour-phase hydrogenation. The quality of the motor fuel is higher and pressure tends to increase the yield, but to prepare heavy oils for vapour-phase cracking an initial hydrogenation in the liquid phase would be necessary in order to prevent coke formation. The highest quality fuels will be produced by high pressure vapour-phase treatment, and the greatest yield will be produced at the lowest temperature at which vapour phase can be induced. R. P.

95. Manufacture of Hydrocarbons. I. G. Farbenind. A.-G. E.P. 315,123, 15.5.29. Conv. (Germany), 7.6.28.

Mineral oils or tars are first subjected to a condensation process and then to hydrogenation in the presence of a catalyst such as molybdenum, zinc and magnesia, at 450° under high pressure. The condensation may be carried out at high or ordinary pressures at elevated temperatures in the presence of catalysts. Gases containing or supplying oxygen, such as steam, are used as condensing agents. Under these conditions lubricating oils are obtained.
T. A. S.

96. Manufacture of Valuable Gases. I. G. Farbenind. A.-G. E.P. 319,657, 31.7.29. Conv. (Germany), 25.9.28.

When carbonaceous materials are subjected to destructive hydrogenation the gases leaving the reaction chamber still contain valuable constituents after the liquid at ordinary temperature has been condensed. The separation of the valuable constituents of the gases leaving the reaction chamber, such as butane, propane, ethane, methane and hydrogen, are dissolved in a washing oil under pressure. The undissolved gases can be used again in a hydrogenating process without further treatment, or may be further freed from hydrocarbons by cooling and compressing.

By releasing the pressure to atmospheric pressure the preponderating portion of gases is again set free, and contains about 60 per cent. of hydrogen and 40 per cent. of hydrocarbons. This mixture is compressed up to 200 to 250 atm. in stages so that between 3 and 9 atm. mostly pentane is obtained, at about 27 atm. butane, and between 80 and 240 atm. propane in the liquid form. The gases leaving the compressor consist mainly of hydrogen, 80 to 90 per cent., with 10 to 20 per cent. of hydrocarbons, mostly methane. By treating under still higher pressures at very low temperatures the constituents can be recovered in a fairly pure state, and the residual gas is suitable for circulating back to hydrogenation processes. R. P.

97. Hydrogenation of Liquids, Solids in Suspension or Gases. Technical Research Works, Ltd., and E. J. Lush. E.P. 336,569, 11.7.29.

Hydrogenation of raw materials by the use of catalysts prepared according to E.P. 203,218 has shown that the activity of the catalyst so prepared is great qualitatively, allowing the hydrogenation reaction to be carried out at lower temperatures than hitherto economically possible. The activity is quantitatively proportional to the anodically oxidised surface of the metallic mass; therefore, it is necessary to obtain the maximum surface and have the metal wire, turnings or plates in such a form as to obtain maximum activity. If the diameter of the cylindrical mass of turnings is greater than 6 ins. the outside of the mass is of greater activity than the inside. The object of the present invention is to provide various forms of compact catalytic mass which will give greater efficiency to the process. Two alternatives are shown wherein the catalytic mass of nickel turnings is packed between two concentric wire gauze cylinders, the diameters of which are, respectively,

18 and 6 ins. A cathode sheet can be used to surround the outer cylinder, and a cathode rod or tube can be placed centrally inside the inner cylinder. Thus the greatest distance that oxidation has to travel from the outside edge nearest the cathode is 3 ins. The metallic gauze cylinders containing the catalyst should be of an alloy which has no deleterious action on the process; for example, the gauze may be composed of copper or monel metal to contain the turnings of pure nickel. R. P.

98. Production of Hydrogenated Aromatic Hydrocarbons. J. Y. Johnson for I. G. Farbenind. A.-G. E.P. 336,583, 10.7.29.

The process consists in treating aromatic materials with hydrogen at temperatures below 350° C. in the presence of mixed catalysts which are immune from poisoning by sulphur or other substances.

These catalysts comprise oxides, hydroxides or sulphides of metals of the 6th group in the periodic system in admixture with metals of group 8 or heavy metals of Group 1 as such or in the form of compounds. Catalyst activating mixtures such as oxides, hydroxides, sulphides, carbonates or oxalates of the elements of Groups 2 to 7 of the periodic system which are not already in the catalyst may be added.

For example, a solvent benzol boiling between 140° and 190° C. is passed at a temperature of 310° C. with hydrogen at 200 atm. pressure over a catalyst consisting of tungsten and nickel sulphides deposited on bleaching earth. The product does not react with concentrated nitric or sulphuric acid, and it is an excellent solvent boiling between 130° and 190° C. The catalyst is prepared by immersing highly active bleaching earth in a normal solution of sodium sulpho-tungstate and allowing it to remain for some time while stirring.

The earth is then filtered off by suction and transferred into a normal solution of nickel nitrate, in which it is stirred for some time. A base exchange takes place in which nickel is fixed by adsorption. The earth is filtered without washing and dried.

Other catalysts are specified for similar reactions. R. P.

99. Destructive Hydrogenation. C. F. R. Harrison, E. D. Kamm and Imperial Chemical Industries, Ltd. E.P. 336,610, 16.7.29.

A process is claimed for the destructive hydrogenation of carbonaceous materials such as coal and oil in which the total products are withdrawn from the reaction vessel, and led to a catch pot maintained at such a temperature that the light and middle oils remain in the vapour phase while heavy oils are condensed. The light and middle oils are removed and condensed for further treatment, while heavy oils are returned to the process. The heavy oils containing solid matter may be passed through a separator to remove solid constituents, and the clean, heavy oil passed back to the reaction vessel.

For instance, a paste of 30 per cent. Bentley coal and 70 per cent. mineral oil is fed into a vertical reaction vessel at a rate of 10 Kg. per hour. The reaction space is packed with tinplated catalyst as described in E.P. 335,215.

Hydrogen is passed into the vessel at a rate of 20 cu. m. per hour. The temperature is maintained at 440° C. and pressure 250 atm. with a contact time of 2 hours. The total products are led to a catchpot in the form of a vertical tower maintained at 350° C. The condensate is withdrawn through a filter and centrifugal separator, and the purified oil is mixed with fresh coal and returned to the reaction vessel. The low boiling products in vapour form are led to a vapour phase process as described in application 21,805/29. The yields are 25 per cent. petrol, 35 per cent. middle oil, 18 per cent. gas, the percentages being reckoned on the amount of solid carbonaceous material introduced into the system. R. P.

100. Production of Hydroaromatic Hydrocarbons. J. Y. Johnson, for I. G. Farbenind. A.-G. E.P. 336,616, 17.4.29.

Hydroaromatic hydrocarbons may be produced from phenols by treatment with hydrogen under pressure in presence of a catalyst and at a temperature of not more than 400° C. above which secondary cracking action takes place.

A large number of catalysts are specified which consist of oxides or sulphides of metals of the 2nd to 7th groups of the periodic system, and preferably of the 6th group in conjunction with copper, silver, gold or metals of the 8th group or mixtures, as metals, oxides or sulphides.

For example, 50 parts of phenol are treated with hydrogen under a pressure of 150 atm., at a temperature of 270° to 300° C., in the presence of 10 parts of a catalyst consisting of cobalt and molybdenum sulphides on active carbon (10 parts of molybdenum and 6 parts of cobalt on 50 parts of carbon). The reaction product consists of cyclohexane and water without any phenol.

The catalyst does not become exhausted even after long use.

Other phenolic compounds such as crude cresol, or B. Naphthol, may be converted in a similar way. R. P.

101. Manufacture of Hydrogen. J. Y. Johnson for I. G. Farbenind. A.-G. E.P. 336,635, 8.4.29.

Natural gas or waste gases rich in methane, such as cracking gases, or gases produced in the synthesis of ammonia, or the hydrogenation of coal, may be converted into gases rich in hydrogen and carbon monoxide by introducing them along with water vapour and air into the charge in a water gas producer, in which coal or coke is undergoing gasification at a temperature of from 800° to 1000° C. In this manner the degasification of the coke takes place, and at the same time the hydrocarbons introduced are decomposed into hydrogen and carbon monoxide. The carbon monoxide may be converted with steam in a known manner into hydrogen and carbon dioxide and the latter removed, thus producing a gas which is mainly hydrogen. R. P.

102. Production of Hydrogen and Gases Containing Hydrogen. J. Y. Johnson, for I. G. Farbenind. A.-G. E.P. 336,944, 8.4.29.

It has been found that the conversion of methane into hydrogen and carbon, or in the presence of water vapour, or oxygen, or both, into mixtures of hydrogen and carbon monoxide by heating to high temperatures may be effected simply and economically when the same is undertaken in one operation with the production of gas by carbonisation of coal.

For example, by leading methane or a gas containing methane and other hydrocarbons, together with water vapour or air through a column of glowing coal undergoing carbonisation, the hydrocarbons are converted into hydrogen and carbon monoxide. A gas mixture is produced which may be worked to yield pure hydrogen by removal of the oxides of carbon or it may be employed without treatment.

Natural gas, or waste gases from other processes, may be used as a source of methane, and any of these gases when passed through a water gas producer may yield hydrogen after the carbon monoxide has been converted with steam into hydrogen and carbon dioxide. R. P.

103. Destructive Hydrogenation. C. F. R. Harrison and Imperial Chemical Industries, Ltd. E.P. 336,991, 19.7.29.

It is already known that destructive hydrogenation can be carried out in a vertical tower, and this type of apparatus lends itself particularly to the use of massive catalysts.

By massive metallic catalyst of large surface area is meant a solid non-granular non-powder metal catalyst of relatively large surface area as distinct from a solid coating or lumps of catalytic material. The expression therefore covers metallic plates, wire, ribbon or gauze.

For example, a number of plates of catalytic material may be assembled side by side to form a unit of square or circular cross section. Such a unit may measure 12 in. vertically and 12 in. in internal diameter, and a tower 20 ft. long may be packed with 16 of these units, placed in such a way that the plates of two adjacent units are at right angles to each other.

The plates may be ¼ in. apart or less. R. P.

104. Conversion of Hydrocarbons into Hydrocarbons of Lower Boiling Point. J. Y. Johnson, for I. G. Farbenind. A.-G. E.P. 337,046, 26.7.29.

It has been found that particularly good yields of products by cracking or destructive hydrogenation may be obtained by employing as catalysts metal compounds, soluble in hydrocarbons, of the type of 1·3 diketones such as acetyl-acetonate or homologues such as chromium or vanadium acetyl-acetonates. These compounds decompose at cracking temperatures and the metals thus set free have a strong catalytic action. The metal constituent of the catalysts is usually a heavy metal, preferably from group 3 to 7 of the periodic system.

It is advantageous to dissolve one of the catalytic substances in the starting material and subject the solution with or without other additions, to heat treatment at ordinary or elevated pressure in the presence or absence of hydrogen or gases containing hydrogen.

Temperature of operation may be from 300° to 600° C., but preferably between 350° and 450° C., and pressures may vary from 20 to 1000 atm. or otherwise.

For example, 2·7 parts of vanadylacetylacetonate and 2·6 parts of nickel acetylacetonate are mixed with 100 parts American gas oil, and the solution hydrogenated at 425° C. and 200 atm. pressure. In addition to gas 31·1 parts of benzene boiling below 180° C. and 57·1 parts of middle oils are obtained. R. P.

105. Process of Treating Hydrocarbons. C. J. Frankforter. U.S.P. 1,780,873, 4.11.30. Appl., 27.8.29.

The process consists of cracking crude oil and distillates in such a manner that the yield of light products is increased, and this result is obtained by subjecting the oil in the process to the action of an activated gas containing hydrogen. The activation of the hydrogen is brought about by heating the gas in contact with a catalyst before introduction into the oil. For example, the gas when subjected to the action of a high resistance electric heating element will be activated and the volatilization of the light fractions will be rapid and complete. The hydrogen so activated will combine with the sulphur compounds in the oil to form hydrogen sulphide and at the same time convert the more complicated sulphur compounds into lighter non-corrosive compounds. Moreover, the hydrogen gas may be passed over a catalysed material to activate the gas to facilitate cracking and eliminate sulphur.

The electric catalytic treatment and the treatment with the catalytic coated igneous material may be combined to prepare the gas, and both processes may be utilised for a definite period, then either may be selectively employed according to conditions.

The apparatus consists of a chamber filled with granular pumice impregnated with a catalyst such as platinum and heated by suitable means. The gas is passed through this chamber, and passes through a tube which extends into the still or cracking retort. The tube (or tubes) is provided with holes or slots on the under side to allow the gas to bubble through the oil. It may also be provided internally with a spiral coil resistance element through insulated leads, in order to further activate the gas. It appears that the electrical treatment is more effective.

In carrying out tests high sulphur crudes lost ⅓ to ½ of their total sulphur as hydrogen sulphide gas. R. P.

See also Abstracts Nos. 33, 134 and 136.

Refinery Plant.

106. Stabilization and Recovery Plant. L. S. Gregory. *Refiner* 1930, **9** (11), 74.

A recovery plant should be designed to extract as much butane from the vapours as it is possible to blend out with the other refinery products to produce a satisfactory gasoline. The recovered gasoline must be stabilized to reduce the propane and lighter content to a negligible quantity. The flow sheet is given of a plant designed to depropinize 1300 brls. a day of Cross pressure distillate, extract 90 per cent. of the butane from the Cross gas and refinery vapours and to stabilize the recovery gasoline.

Hot vapours from the Cross tower are partially cooled and the condensate, practically free of propane and lighter, is withdrawn from the base of the accumulator and cooled. The hot gases are further cooled and introduced into the Cross separator. The cool gas from here goes to the recovery plant, while the cool liquid passes to a surge tank. From here the liquid passes through a heat exchange with the hot bottoms and thence to the P.D. stabilizer. The overhead vapours from the stabilizer are water cooled and the condensate pumped back as reflux. The base of the column is steam heated, and the hot bottoms, which are the stabilized pressure distillate, are passed through a heat exchange and then water cooled.

Gases are passed through one stage of compression and cooled in the interstage cooler. Condensate is pumped through heat exchange with hot bottoms to the gasoline stabilizer, while the gas is high staged and sent directly to the stabilizer.

The economy in the installation lies in the fact that a large portion of the pressure distillate, that does not require stabilizing, is removed, and that the temperature of the base of the P.D. stabilizer is considerably lowered by the removal of these heavy ends.

In the recovery plant extraction and stabilization take place in the same column. Considerably less equipment is required compared with an oil absorption plant and gasoline stabilizer, and consequently the initial cost is lower. W. L.

107. Catalytic Gas Reactions. Holzverkohlungs-Ind. A.-G. E.P. 335,411, 23.10.29.

A description is given of a reaction chamber for catalytic reactions in the gaseous state, having two of its walls parallel and separated by a distance which is small compared with the other dimensions of the reaction-chamber.

It is claimed that good heat transference is achieved, due to the flat nature of the reaction chamber, and the necessity of arranging a large number of tubes, of small diameter, in parallel, may be thus avoided. Containers up to 100 l. capacity are mentioned. E. N. H.

108. Regulating Valves for Controlling Fluids under High Pressure. W. Pfeiffer and K. Reiss. E.P. 337,838, 7.8.29. Conv. (Germany), 18.5.29.

According to this invention there is provided a regulating valve for controlling fluids under high pressures and temperatures in which the packing of the valve spindle is effected by a membrane mounted remote from the valve. The membrane is in the form of a longitudinal resilient metal tube of phosphor bronze, aluminium or the like located between the valve and a hand wheel.

The operation of the valve cone is effected by long rods which are guided in a straight tube capable of withstanding the operating pressure. By means of a suitable length and small cross section of the rods and the guide pipe, heat conduction is minimised. At the outer end of the guide pipe the packing member may be soft soldered. In order to avoid any possible penetration of hot vapours to the soldered joint, care is taken that the guide pipe is always filled with liquid.

As a sealing member, a seamless tube of phosphor bronze or aluminium is suitable, as it will, with a clear diameter of 6 mm., stand a pressure of 300 to 400 atm., provided there is no lateral movement. This may be ensured by carrying it on a mandrel, which is polished to prevent scratching or rubbing by the frequent to and fro movement of the tube. The mandrel is operated by the hand wheel. R. P.

109. Treating Hydrocarbon Oils. C. W. Watson. U.S.P. 1,780,286, 4.11.30. Appl., 1.7.27.

This invention relates to a process of fractional distillation which is particularly adapted for the distillation of petroleum, in a battery of stills fitted with fractionating or rectifying means. The stills are preferably connected in series, and arranged so that they can be heated successively to yield distillates of consecutively increasing boiling points. The vapours evolved from the stills are subjected to fractionation, and the vapours are condensed and collected from the first several stills of the series, whereby a distillate fraction is formed which contains the more volatile constituents of the oil charge. The charge may be divided into a larger and a smaller fraction, the smaller portion being introduced as a reflux medium to those towers corresponding to the stills from which vapours composing said distillate fraction are obtained, and introducing the larger portion of the charge into the first still of the series. H. E. T.

110. Steam Distillation. E. H. Lealie and E. Baker. U.S.P. 1,780,977, 11.11.30. Appl., 11.5.23.

A method and apparatus are described for fractionating a petroleum oil and recovering its various fractions. The mineral oil is fractionated in a column of superimposed sections containing packing material and allowed to flow downwards so as to come into intimate contact with the ascending oil vapours. The condensed vapours and refluxing condensate leave the top of the fractionating column and a liquid product can be withdrawn independently from any desired section, a conduit connection being made to one or more sections for delivering a liquid to the sections. Part of the liquid residue leaving the lower end of the column is vaporised by further

introduction of steam, and the resultant vapours are returned to the lower end of the column. The final liquid residue remaining from the steam distillation is further heated and subjected to flash-vaporisation, and the resultant vapours as before are introduced at the bottom of the column.

W. S. C.

See also Abstract No. 25.

Analysis and Testing.

111. Low Temperature Oxidation: Part III., The Lag in Ignition of Some Hydrocarbons. J. S. Lewis. *J.C.S.*, 1930, **131**, 2241.

A consideration of the methods of determining the lag in ignition is followed by the adoption of a modification of the drop or crucible method in which the time between the fall of the liquid drop and the appearance of flame is measured. Details are given of the precautions necessary to obtain duplication. A phenomenon of this method is the fading away of spontaneous ignition at some particular temperature dependent upon the molecular weight of the paraffin, the dimensions of the tube or bulb, and the activity of the oxidising agent present. This appears to be another case of flameless combustion, and forms a serious objection to the use of the crucible method, although it would appear that the ignition fading temperature is a measure of the oxidisability of these hydrocarbons.

Following upon a criticism of the drop method as a test for petrols, the conclusion is drawn that the ignition temperature of a petrol, determined by this method, cannot be utilised as a measure of the knock rating because it does not vary sufficiently with the presence, or absence, of the so-called heavy ends.

The effect of catalysts has been considered. Substances which promote oxidation have been shown to raise the ignition temperature and the time of lag. In some cases, however, by reducing the proportion of certain of these substances to minute amounts and raising the temperature, the time of lag has been diminished. When anti-knock reagents are present the rates of change of lag with temperature are far less than the rates produced by the promoters of oxidation, and a reduction in the time of lag is not obtained by decreasing the quantity of catalyst. When members of each class are present together, they tend to neutralise each other's effect.

E. N. H.

112. Resistance Thermometers for Chemists. C. G. Maier. *J. Physical Chem.*, 1930, **34**, 2860–2868.

Advantages of copper for resistance thermometers are pointed out, and a type is described which eliminates hysteresis and drift due to differential thermal expansions. A method is given for the precise calibration of a copper resistance thermometer from two accurate fixed points and two other coefficients of less accuracy determined with a mercury in glass thermometer. The precise thermometric points utilised were that of melting ice, 0° C., and the sodium sulphate decahydrate transition, 32·384° C.

Between 0 and 32·4° C. the resistance of a thermometer constructed of commercial copper wire was found to obey the parabolic expression:—

$$Rs = Ro\,(1 + 4.26399 \cdot 10^{-3}_{s} + 3 \cdot 753 \cdot 10^{-7}_{s^2},$$

where Rs is the resistance at temperature s, and Ro that at 0° C.

A modified type of resistance thermometer suitable for general chemical laboratory use is described, which is the practical equivalent of several hundred multiple junction thermals, and which requires only simple electrical instruments. Since the resistance thermometer of the modified type can be made to have zero potential at any desired temperature, and since the direction of the potential changes sign at this temperature, this arrangement has interesting possibilities as a thermo-regulator operating without mercury, without make and break contacts in the regulator, and with practically negligible mass and heat lag. Experiments with such use are in progress.
W. S. C.

113. Determination of Evaporation Losses. G. W. Chenicek and W. G. Whitman. *Oil and Gas J.*, 1930, **29** (26), 78, 263.

The vapour-pressure method offers a simple means for determining a property of gasoline which is sensitive to slight losses by evaporation. The method permits direct comparison between two samples without further experimental data and can, therefore, be applied on any type of gasoline. Measured amounts of dry air are bubbled through a sample of gasoline at standard temperature in such a manner as to ensure practical saturation. The resulting vapour volume is determined and the decrease in liquid volume is also determined. Simple calculations then permit the computation of the vapour pressure of the sample. Successive determinations are made on the same gasoline until about 5 per cent. has been evaporated, and the data is represented on a semi-log plot of vapour pressure *versus* percentage evaporated. As the operation requires only the collection of samples and subsequent laboratory determinations, it can be employed in many conservation problems where accurate gauging or vapour analyses are impracticable. Theoretical consideration of its inherent accuracy are confirmed by the data here presented on field and laboratory tests.

The storage tests here reported are also interesting as a confirmation of the results on unprotected tanks, and as an indication of the high efficiency of breather bags for protecting standing storage. The displacement tests demonstrate the effectiveness of floating roofs on working tankage, but show that evaporation losses due to rapid filling of standard roof tanks in good condition are not as high as has been generally assumed.

The present accuracy of about 0·04 per cent. is adequate for the detection of small losses, and is greater than would be required in many cases.

Considering this accuracy, and the wide field of applicability, it appears that the vapour-pressure method may be the most generally satisfactory means at present available for studying evaporation losses. W. L.

Road Construction.

114. Australia's Effort to Solve Road Problems. (Light Bituminous Wearing Coats.) J. R. Bathgate. *Roads and Road Constr.*, 1930, **8**, 436–439.

Possibilities of economic achievements and the conservation of old roads are afforded by the use of such coats. There is no rule of thumb method for tar or bitumen surfacing of penetration pavements and spraying of macadam and gravel pavements.

Experiments with surfacings are outlined. The use of light grade bitumens (200 penetration) and Nos. 2 and 3 medium grade tars, and priming tars from Crude to No. 1 grade in the main, led to failure. A section of old gravel road resheeted was lightly reinforced with 1½ and ¾ in. gauge slag.

A prime coat of No. 2 tar and ¼ in. slag was followed by 60 to 70 penetration bitumen with a thinning agent and ¾ in. slag was well rolled in. Thickness of wearing coat 1 in. Total cost 2s. 11½d. per sq. yd. This road has withstood heavy traffic satisfactorily for two years, probable life four years. Probable overall cost should not exceed 1s. per sq. yd. per year.

Good results were obtained from a prime coat of heavy grade tar fluxed with selected heavy tar oil followed by ¾ in. slag and a sealing coat of No. 3 tar and ¾ in. slag. The use of a bitumen primer and seal was considered well worth the extra expense. Other work involving liquid bitumens and road oils led to the belief that one direct coat of bituminous surfacing is equal to two coats of tar surfacing. The advantages and disadvantages of the use of cold application emulsions are also given. C. I. K.

115. The Highways of the U.S.A.: Current Constructions, Types, Methods and Costs. T. Warren Allen. *Roads and Road Const.*, 1930, **8**, 394–396, 431–433.

Part I., Finance and Road Types.—Details are given of the bodies responsible for rural highway development in the U.S.A., the statistics for total miles and percentages of each type of road (17 per cent. of the surfaced mileage are surfaced with a high type penetration macadam or better) and the funds available for the work.

The use of untreated gravel, top soil and sand clay for road surfaces is a desirable means of securing all-weather highway service at low cost. However, this fails to offer a complete solution of the provision of adequate highway service. Where possible, roads of the higher type are recommended on account of their all-the-year-round usability.

Part II., Surface Treatment of Gravel and Other Roads.—The need for surface treatment of sand, clay, top-soil and gravel surfaces led to the development of bituminous material having an improved dust penetrating power. Details are given of the construction methods for low-cost roads, involving the preparation of the surface for the application of bituminous material having dust penetrating power followed by a second bituminous treatment for the production of a mat. The Indiana retread method of treating old road surfaces and gravel surfaces to carry heavy traffic is outlined. C. I. K.

116. Asphaltic Material. G. Abson, Assr. to Chicago Paving Laboratory. U.S.P. 1,782,186, 18.11.30. Appl., 25.10.29.

The patent describes means whereby petroleum residue may be converted into blown asphalt having a higher penetration for a given melting point than can be obtained from the same residuum without treatment. A small quantity of an inorganic non-alkaline metallic salt (*e.g.*, iron or zinc chloride) which is non-volatile at blowing temperatures is incorporated with the residue. The quantity of salt should be insufficient to effect cracking at blowing temperatures but sufficient to act as a condensation agent. The residue, with its suitable admixture of metallic salt, is then air blown to the specified melting point. W. H. T.

Geology.

117. Origin and Environment of Sources of Sediments of Petroleum Deposits. J. D. Haseman. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1465–1468.

The writer claims to have discovered an actual case in Florida of the formation of asphalt and hydrocarbons from humic acid in swampy conditions in a small fresh-water lake fed by tributaries rich in humic acid. He

claims that hydrocarbons are synthesized from the humic acid by the activation of silicon—and carbon—containing materials which produce the necessary chemical relations at ordinary temperature by means of specific hydrogenions.
V. C. I.

118. Does Petroleum Form in Sediments at Time of Deposition? Parker D. Trask and C. C. Wu. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1451–1463.

Selected bulk samples from typical sediments were extracted with CCl_4 to examine for hydrocarbons. The material examined included samples from Florida Bay, the Californian Coast, Pimlico Sound and an Agal Lake in Florida. The extracts include small quantities of solid paraffins, traces of fatty and oily substances, also fatty acids and quantities of organic sulphur compounds. The results are claimed to indicate that liquid petroleum is not present during deposition, save perhaps in infinitesimal quantities. The author concludes that most of the petroleum must be obtained from the non-fatty organic matter which is entombed in the sediments.
V. C. I.

119. Geology and Mineral Resources of Parts of Carbon, Bighorn, Yellowstone and Stillwater Counties, Montana. R. S. Knappin and G. F. Moulton. *Bull. U.S. Geol. Surv.*, 822A, 1930.

The exposed sedimentary rocks in this area range from the Mississippian to the Quaternary with frequent erosional disconformities, the Cretaceous being particularly well represented. The structure is a badly faulted plunging anticline. Although the area contains abundant carbonaceous shale and many loosely cemented sands that might act as reservoir rocks, the structures appear to be relatively unfavourable. The absence of salt water in the wells suggests complete flushing, and the results of drilling have been, up to the moment, discouraging.
G. R.

120. Salt Flat Oil Field, Caldwell, County Texas. L. F. McCollum, C. J. Cunningham and S. O. Burford. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1401–1423.

This field was discovered 2 years ago and lies 4½ miles south-east of Luling oilfield. It is located against a fault which strikes N 30° E. and lies on the south-eastern or upthrown side. The maximum displacement of the fault is 375 ft. The exposed rocks belong to the Wilcox beds. The oil formation is in the upper portion of the Edwards Sst., which is porous and dolomitic. The general structure includes a tendency to form slight anticlinal conditions close to the fault plane, and the area of production is largely confined to this zone, suggesting that the structure is either a faulted anticline or a faulted monocline with reverse drag near the fault plane. The estimated total recovery from the field is 36,000,000 brls., giving an ultimate approximate recovery of 36,000 brls. per acre.
V. C. I.

121. Petroleum Potentialities of Gulf Coast Petroleum Province of Texas and Louisiana. Donald C. Barton. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1379–1400.

The extensive geophysical work in the Gulf Coast province in the last few years has led to a considerable areal extension of petroleum potentialities in Texas and Louisiana. The salt dome area now stretches as far east as the Mississippi river and westwards almost to the Rio Grande. Further, there has been a very important increase in the depth of production, which has now extended to 7440 ft. and ranges in age from the Eocene to the Pliocene.

About 55 per cent. of the oil is obtained from Miocene formations, 35 per cent. from the Oligocene and the rest from the Pliocene and Eocene. Deeply buried salt domes overlain by great thicknesses of strata are being discovered, but the drilling development of such an area is naturally slow in view of the uncertainties of the structure. This great extension both in depth and area of the petroleum province has increased very considerably the recoverable reserves of oil. The author considers that the former estimate of 2,300,000,000 brls. is now too low, and estimates the present reserves at a minimum of 3,500,000,000 and a possible maximum of 10,000,000,000 brls. V. C. I.

122. Geology of Some Salt-Plugs in Laristan (Southern Persia). J. H. Harrison. *Quart. J. Geol. Soc.*, 1930, **86**, 463–522.

This paper deals with the geology of salt-plugs found in Laristan, and contains a description of 51 individual occurrences—in many cases accompanied by small sketch maps. The sediments range in age from Ordovician to Recent, and the salt-plugs occur penetrating the crest of anticlines, though they are found almost as common at the plunging end of folds. They have not been intruded simultaneously, but appear to have been associated with movements in the area since mid-Cretaceous times. Comparisons are made with salt-intrusions of Rumania and Germany and elsewhere, which resemble topographically those of the Persian salt-plugs.

The author considers that they have undoubtedly been over-ridden and then pressed out at the surface as extrusions. The force which drove the salt to the surface is not clear. The effect of load must have been the chief factor where the plug emerged through strata that were flat or gently warped. Beds of 9000 ft. and 19,000 ft. have been penetrated in the case of Cretaceous and Pliocene salt-plugs respectively. To drive a plastic mass, as salt or gypsum, through such a thickness of deposits, very great pressure must have been operative. G. S. S.

123. Mexico's Future Petroleum Possibilities. Jack Logan. *Oil, Weekly*, 1930, **59** (12), 27–42.

In this paper (which is accompanied by a map and well information) the geological structures of the concessions in the States of Nuevo Leon and Tamaulipas are described. The deposits are mainly Cretaceous in age, though to the east these are covered by Lower Tertiary sediments. Some of the best known structures of North Mexico occur in the area—*e.g.*, the great plunging anticline (" Salado Arch "), the Tanque-Barreta, La Lajilla, Guerrero Dome, and other anticlines are discussed individually.

The region is considered as one possessing vast possibilities of oil production, and consequently the coming years will bring more drilling in North-eastern Mexico than the district has hitherto witnessed. G. S. S.

124. Geophysical Surveys in Oil Territories. J. B. Ostermeier. *Oil News*, 1930, **28**, 546.

Success has been attained, where oil occurs in association with salt deposits both by gravimetric and seismic surveys, but direct location of the oil cannot be given with any degree of reliability.

Results obtained by electrical surveys denote differences of conductivity over oilfields, and Friedl has referred to these surveys as giving remarkable results.

Magnetic measurements offer further possibilities of disclosing tectonic details by the use of highly sensitive instruments. The determination of synclines can be accomplished, according to the author, quite successfully by recording the maximum disturbance exactly above the syncline.

The indications appear to increase with the depth of the syncline, and this fact is of particular importance to oil prospecting. L. W. C.

Engines.

125. Testing Supercharged Engines. J. Pettitt-Herriott. *Aircraft Eng.*, 1930, **2**, 300–304.

Particulars are given of corrections to be applied to results of tests at ground level, on engines fitted with gear driven centrifugal supercharger blowers, to obtain comparative engine performance at specified altitudes.

The following terms are defined: (*a*) Rated altitude; (*b*) rated boost pressure; (*c*) rated full power; (*d*) ground level power; (*e*) maximum permissible boost pressure; (*f*) standard atmospheric conditions.

The apparatus used is described and illustrated. The main feature is the altitude depression box connected to the air intake of the supercharger. Holes in the top and bottom of this box can be partially or completely closed by rotating shutters, thus simulating altitude conditions. Four mercury manometers register the pressure in: (1) The altitude depression box; (2) boost gauge connection on engine; (3) inlet supercharger impeller; (4) the exhaust back pressure in the silencer system of the test bench.

The methods of calculating and applying the corrections for the following observations are given together with a worked example: (*a*) Exhaust back pressure; (*b*) compression ratio of supercharger blower; (*c*) rated boost pressure; (*d*) observed h.p. for rated boost pressure; (*e*) h.p. at rated boost pressure; (*f*) ground level power for the effect of air intake temperature at altitude Z.; (*g*) h.p. for the difference in charge and exhaust pressure at altitude Z; (*h*) rated full power.

Approximate methods of calculation are also included for rated boost pressure and rated full power together with charts for obtaining: (1) Corrected boost pressure; (2) h.p. at the rated boost pressure; (3) ground level and rated full power.

For supercharged engines the power and throttle curves of normally aspirated engines are substituted by a rated altitude power curve and a constant boost throttle curve. The construction of these curves is outlined.

C. C.

126. Present Status of Aircraft Superchargers. S. Paul Johnson. *Aviation*, 1930, **29**, 358–359.

The supercharger provides sea-level engine power at a high altitude, and can increase output under sea-level operating conditions. The limits to supercharging are detonation and general engine durability. Commercial superchargers fall into two classes: (1) Centrifugal compressor type (highly developed rotary air compressors, exhaust driven, which are connected to the engine through the exhaust manifold and carburettor air intake only). The ratio of crankshaft speed to impeller speed ranges from one to five up to one to fourteen. The high velocity air from the impeller goes through a diffuser where energy is converted into pressure. (2) The roots blower or positive pressure variety (The De Palma), mounted as a separate unit and gear driven (see National Advisory Committee for Aeronautics Tech. Report, No. 230, 355).

Diesel engines can have their power output increased by 40 per cent. by supercharging.

C. I. K.

127. Design and Manufacture of Marine Auxiliary Oil Engines. O. Wans. *Mar. Eng.*, 1930, **53**, 449–452.

For powers up to 1200 h.p. the four-stroke cycle engine is favoured. The reliability of an engine depends on the mean effective pressure, and the piston and rotational speeds. The number of power cylinders has a direct

bearing on engine balance, the resulting vibration and reliability. Two and four cylinder engines may set up forces which may cause severe vibration. Should this synchronise with the free period of oscillation of the hull, serious damage to engine or hull may ensue. Five cylinder engines fitted with balance weights are satisfactory. Torsional effects can be suppressed by dampers (Lanchester type), and combined stiffness of crankshaft, bedplate and housing is essential to reliability. Details are given of the design of a five cylinder 500 B.H.P. engine. The lubrication systems (which affect reliability) comprise a hand pump for flooding bearings before starting, after which a pump, gear-driven from the crankshaft, maintains a pressure of 10–25 lb. per sq. in. in the forced feed system. Satisfactory results demand an oil cooler, an oil filter (types are described), and an ample quantity of oil in circulation. C. I. K.

128. Benes Diesel Engine Fuel Injection Pump. *Mar. Eng.*, 1930, **53**, 460–461.

A complete description with diagrams is given of the construction and method of operation of this fuel pump which is suitable for high speed engines. Accurate metering and a rapid destruction of pressure (which ensures rapid closing of valve and no drip afterwards) are claimed. Air Ministry trials are being carried out. C. I. K.

129. Fuel Problems in the Mercantile Marine. W. J. Muller. *Mar. Eng.*, 1930, **53**, 465–467.

Fuel problems are made up of fuel price, reliability and cost of upkeep and repairs and "personnel." Low oil fuel price initially encouraged oil firing, but high prices during the war in some cases caused a reversion to solid fuel. The use of oil fuel influenced the type of boiler used for steam generation, the Scotch boilers being replaced by water tube boilers which have large combustion chambers suitable for firing with oil or long flaming high volatile coal.

The Underfeed mechanical stoker, enlargement of combustion chamber space, etc., raised efficiency of water tube boilers to 75 per cent. on coal. The question of using cheap high ash coals of small particle size, high volatile coals and liquid fuels are dealt with.

The Lentz standard marine engine (poppet valves) was preferred to others, as it required less lubricating oil for the cylinders. The fuel problems of Diesel engines were experienced with the first Diesel-engined ships. Though light diesel oil (solar oil) is more expensive than ordinary diesel oil, its use in Sulzer Diesel engines gave best results. C. I. K.

Coal and Shale.

130. The Evaluation of African Torbanite. P. Niemann. *Brennstoff-Chem.*, 1930, **11**, 469–471.

Torbanite is described as consisting of geologically old sediments which are intermediate in chemical composition between bituminous shale and lignite.

In the present research two different types of torbanite—namely, Transvaal and Angola torbanites—were investigated. One ton of the sample was utilised for the research.

The Transvaal torbanite was a dark brown, hard, solid, apparently homogeneous, but actually had a shell-like structure. The crude substance gave considerable yields of useful products. The tar was poor in paraffin and on

distillation gave a coke which could be used for electrodes on account of its high carbon content. The residue from the coke distillation contained 27·6 per cent. of fixed carbon. The distillation water reacted alkaline and had an ammonium content of 0·15 per cent.—*i.e.*, equivalent to 8·15 kgm. of ammonium sulphate/ton of torbanite. This amount is 20 per cent. greater than that obtained from Scottish shales. Acid and lye treatment of the by-products gave good technical products. The following yields were obtained by distillation of the tar and subsequent treatment of the oil :—

100 kgm. of tar yielded..	kgm./ton of torbanite (with 1% moisture)
9·60 kgms. fuel to 150°	34·08 kgms.
8·00 „ heavy benzine (150–200°)	28·40 „
14·40 „ motor oil	51·12 „
33·15 „ paraffin oil	117·68 „
11·00 „ machine oil	39·05 „
3·25 „ paraffin	11·54 „
12·60 „ coke..	44·73 „
8·00 „ gas + loss..	28·40 „
100·00 kgms.	355·00 kgms.
Ammonium sulphate	8·15 kgms.

The torbanite from Angola was different from the previous sample in appearance and chemical properties. It had a black fractured surface of high specific gravity. The distillation water from the steam distillation reacted alkaline, the ammonium content amounting to 0·12 per cent. The crude substance consisted of only small quantities of light-boiling oils and gave good yields of higher-boiling constituents from the tar. Only traces of paraffin were obtained from the tar ; the coke from the tar distillation consisted of pure carbon which was valuable for electrodes. Acid and lye treatment of the by-products gave good technical products which could be utilised as fuel oils and asphalt.

The composition of the Angola torbanite is given in the following :—

100 kgms. tar yielded	kgm. / ton of torbanite (with 4% moisture).
0·3 kgms. fuel to 150°	0·72 kgms.
3·3 „ heavy benzine (150–200°)	7·92 „
45·4 „ gas oil	108·96 „
31·5 „ machine oil	75·60 „
11·0 „ coke	26·40 „
8·5 „ loss	20·40 „
100·0 kgms.	240·00 kgms.
Ammonium sulphate	7.45 kgms.

W. S. C.

131. Coal and Oil Fuel Problems. S. B. Freeman. *Mar. Eng.*, 1930, **53**, 467–468.

Details are given of results obtained with coal, boiler oil in Scotch boilers and Diesel oil in Diesel engines. With oil fired boilers, it is useful to have carbon dioxide recorders, and recorded temperatures of the funnel, as a means of controlling the burners and a check on excess air. The use of a higher grade Diesel oil leads to fewer repairs to machinery, and such oil should be centrifuged before entering the cylinder if possible. C. I. K.

132. The Determination of Acid Constituents in Shale-Oils and Their Residues. E. v-. Peyold. *Chem. Zeit.*, 1930, **54**, 678.

Six methods for the determination of acid constituents in shale-oils are described together with the results of tests made with each method. It is concluded that the best method is that of Holde, although certain defects are apparent. A modification of the Marcusson titration method may be employed for the determination of organic acids in shale-oil products. An aliquot part (5 c.c.) is titrated with N/10 alcoholic caustic soda solution using alkali-blue as an indicator, the remainder of the acid solution being then neutralised with the calculated quantity of N alcoholic caustic soda solution. It is pointed out the method is advantageous in that the change of colour is easily observed and that a smaller amount of indicator is required. Phenols and like compounds are not titrated. W. H. T.

133. Hydrocarbons of High Boiling Point Range from Coal, Shale and Other Bituminous Substances. I. G. Farbenind. A.-G. E.P. 312,050, 8·4·29.

A process is described for the production of hydrocarbons of high boiling point range, such as lubricating oils from bituminous substances, such as coal, shale or other solid bituminous carbonaceous materials, which consists in treating the initial materials with organic solvents at an elevated temperature and under pressure, and then preferably directly subjecting the products extracted to a treatment with gases having a reducing action, under such conditions of temperature and pressure and only for such a period of time that no extensive decomposition into products of lower boiling point occurs. The reducing gas is preferably hydrogen, and if desired paraffin wax may be separated off before the reduction treatment. H. E. T.

134. Separation of Oils, Tars and the Like from Solid Constituents. I. G. Farbenind. A.-G. E.P. 312,228, 1.5.29. Conv. (Germany), 22.5.28.

The separation of oils, obtained in the treatment of various coals, tars and mineral oils with hydrogen under pressure, from the solid residues, is frequently difficult, but can be effected in an advantageous manner by mixing with sawdust, brown coal ash or light ash from flue gases, and centrifuging or filtering the mixture. In part the residues are of a colloidal character which is destroyed by the addition of these materials so that separation may be effected in a fraction of the time required without their addition.

For example, 50 kg. of residue containing about 40 per cent. solids obtained from the destructive distillation of brown coal are stirred with from 10 to 15 per cent. of sawdust or light ashes which have been deposited from flue gases. After heating to about 110° C. and dilution with 50 kg. of heavy benzine, the whole is stirred for about 5 min. at 110° C. and filtered. The reduction of time necessary for filtration in contrast to that without additions amounts to 85 per cent. R. P.

135. Manufacture of Liquid Products from Solid Distillable Carbonaceous Materials by Destructive Hydrogenation. I. G. Farbenind. A.-G. E.P. 315,764, 15.7.29. Conv. (Germany), 17.7.28.

Carbonaceous material such as brown coal is subjected to a mild hydrogenation under pressure, so that the formation of benzine and consequent precipitation of asphaltic matter is avoided. The material is passed through the hydrogenation apparatus countercurrent to the hydrogen at about 450°

and 200 atm. From the gas a light oil is condensed which on further hydrogenation gives benzine. The solid or semi-solid residue is extracted with benzol under a pressure of about 75 atm. The extracted materials are of high molecular weight and can be worked up into lubricating oils.

T. A. S.

136. Hydrogenation of Coal. Imperial Chemical Industries, Ltd., R. Holroyd and C. Cockram. E.P. 337,028, 18.7.29.

The invention relates to the hydrogenation of bituminous coals, and consists in a process of more than one stage wherein a preliminary treatment in presence of hydrogen at or near the temperature of decomposition is followed by destructive hydrogenation.

The first stage is conducted under such conditions that the formation of phenols is prevented or reduced in the subsequent stages. Preferably all stages are carried out in the presence of oil and catalysts.

Coals which have been heated up to or just above the decomposition temperature possess increased activity; they are more easily oxidised and are more easily dispersed in pyridine and the like.

For example, Bentley coal is mixed with heavy oil obtained from a later stage of the process and treated with hydrogen at 250 atm. The temperature of initial decomposition is 390° C., and the reaction temperature is therefore maintained at 390° C. within 5° or 10°. The catalyst used is sheet iron coated with tin. Little cracking takes place and little or no gas is formed, whilst the coal is converted into a pitch-like material soluble in chloroform and similar to a hydrocarbon.

The oxygen of the coal is removed by a reaction which appears to be a true reduction and not destructive hydrogenation. As much hydrogen as possible is added without cracking, about 4 per cent. by weight.

The fusible material obtained in the first stage is treated with hydrogen at 250 atm. pressure and 460°–470° C. for one hour in presence of sheet iron coated with tin. The product consists mainly of petrol, middle oil and heavy oil.

In the third stage the residual middle oils and heavy oils are again hydrogenated and part of the heavy oil is used in the first place.

R. P.

137. Retorts for the Distillation of Coal, Shale, Peat and Other Carbonaceous Substances. T. Topping and J. S. Black. E.P. 337,842, 27.3.30.

This invention relates to the low temperature continuous distillation of coal, shale, peat and other carbonaceous substances in improved vertical retorts in which cross flues are arranged, the bottom of the retort being sealed whilst discharging. A preliminary drying of the material in the hopper is brought about by means of a flue surrounding its lower portion, which is connected with the flue surrounding the retort as well as with a chimney or exit. A depending sleeve, whose base is at a lower level than the gas outlet part from the retort, forms a continuation of the bottom of the hopper and serves as a means for preventing the material passing from the hopper to the retort obstructing the gases passing off from the retort.

W. S. C.

138. Freeing Tars and Oils from Solid Particles Contained Therein. J. Y. Johnson. E.P. 338,154, 14.5.29.

A process is described for the separation of oils obtained by the destructive hydrogenation of various kinds of coal, tars, mineral oils and other similar mixtures from suspended solid residues. It consists in passing through

the mixture gases exerting an acid reaction against litmus when dissolved in water and which do not undergo oxidation or reduction during the treatment. The mixtures are then filtered or centrifuged. In particular, the passage of a stream of CO_2 or SO_2 through the mixtures to which a solvent —*e.g.*, benzine—has been added is found very suitable. The temperature at which the treatment is carried out should, in any case, be so high that the material has sufficient fluidity. The treatment need only be of short duration, the flocculation, conglomeration, and increase in the size of the particles effecting a considerable reduction in the time necessary for filtration and centrifuging. W. S. C.

139. Neutralising Washed Light Oil. J. J. Lawton, Assr. to Semet-Solvay Co. U.S.P. 1,779,944, 28.10.30. Appl., 31.12.27.

The invention relates to the refining of crude light oil recovered from gases produced during the destructive distillation of coal, particularly to the neutralising of such oil after treatment with sulphuric acid. The acid-washed oil is treated with ammonia to neutralise residual acid and form a neutralisation liquor. Low boiling constituents are separated from the neutralised oil by direct steam distillation, while maintaining an alkaline condition in the still by the presence therein of the neutralisation liquor. The decomposition and volatilisation of any sulphur compounds present in the neutralised oil is thus prevented. W. H. T.

See also Abstracts Nos. 99 and 101.

ABSTRACTS.

Geology.

140. Subsurface Distribution of pre-Mississippian Rocks of Kansas and Oklahoma. H. W. McClellan. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1535–1556.

The paper deals principally with the geology of the pre-Mississippian rocks of Kansas but in the two maps appended the results are extended to include data for Oklahoma and also North Texas. The principal pre-Mississippian formations are described and their distribution underneath the Mississippian formations is tentatively given on a subsurface geological map. The nature of the unconformities of the overlying Mississippian and Pennsylvanian are described and the broad features of the pre-Mississippian structures are delineated. It is shown that the present distribution of the rocks is the result mainly of two important earth-movements which occurred respectively between Devonian and Mississippian and also in post-Morrow time. The interformational unconformities were in some cases extensive, but gentle erosion appears to have progressively increased towards the north-west. V. C. I.

141. Oklahoma City Oil Field, Oklahoma. H. H. Charles. *Bull. Amer. Assoc. Petr. Geol.*, 1930, **14**, 1515–1533.

The surface structure of the Oklahoma City Field consists of an asymmetrical anticline in Permian rocks approximately 9 miles long and 4 miles wide with a steep north-easterly flank. Below the exposed Permian formations the underlying Pensylvanian series is strongly faulted on the north-eastern flank of the structure, and the substructure of the fold consists of the faulted anticline with a gentle south-westerly dip and a down-thrown fault on the north-east flank.

The Pennsylvanian rests on the underlying formations with marked unconformable overstep; this has resulted in the elimination of the Mississippian formations from the crest of the fold where the Pennsylvanian rests directly on the eroded Arbuckle Limestone.

Some oil has been noted in the Pennsylvanian series and also in the underlying Simpson formation, but the most important oil horison lies within the Arbuckle Limestone where big production has been obtained at depths approximating 6,000 ft. The most important oil horizon lies from 200–500 ft. below the top of the Arbuckle Limestone. V. C. I.

142. Mexico's Future Petroleum Possibilities. J. Logan. *Oil Weekly*. 1930, **59** (13), 31–34; 1931, **60** (3), 32–34, 87, 88.

The first article deals with the State of Coahuila, in the northern portion of which numerous geological structures favourable for oil accumulation are known to exist. The rocks are of Cretaceous age, represented by the Comanchean and Glenrose Limestones, the former being the most extensive and more folded than the same formation north of the Rio Grande. The Glenrose Limestone shows considerable oil

saturation, which, according to geologists familiar with North Coahuila, should be productive under favourable structural conditions, and possibly deeper production may be had from Permian and Pennsylvanian rocks.

An extensive trend of folding occurs from the Rycade Field, through Jiminez, Coahuila and onwards for at least 100 miles. Other existing folds referred to are those of the Esperanzas and San Marcos anticlines. A well-boring at the latter (25 miles south of Cuatro Ciebegas) is stated to be of considerable geological interest, being the lowest horizon yet tested in Mexico. Its location lies several hundred feet below the base of the Tamasopa Limestone and there are possibilities also of penetrating the Pennsylvanian rocks.

The second article, the last of a series, deals with the Guerrero Dome, which is about 8 miles long and 3 miles wide and trends almost due north and south. The fold is an anticline of the Rocky Mountain type, with two domes at the top of the anticline separated symmetrically by a central saddle. In drilling, the top of the Medway was found at 2,287 ft., and so far over 4,900 ft. of this formation has been penetrated; the depth of the well at present being over 7,000 ft.

Though the base of the Medway and the top of the Papagayos (Up. Cretaceous) have been virtually reached, there is no assurance that either will be found productive. The main production of Mexico—the Tampico districts—was from the *Lower* Cretaceous, which apparently will be too deep at Guerrero to attempt to reach. There are, however, structural possibilities that production may be found on the south dome flanks, or in wells drilled on the crest or flanks of the north dome.

G. S. S.

143. Hobbs Area Presents Many Geological Problems. F. Swindell. *Oil Weekly*, 1931, **60** (3), 25–26, 90.

Hobbs Pool is located near the eastern edge of the Permian Basin in New Mexico. Quaternary caliche overlie thick Triassic red beds, which are terrestrial and contain no oil. The brown lime, a guide horizon in the Permian, is met at 2,730 ft. to 2,945 ft., and 400 ft. below this is the Bowers sand with the first shows of oil. The main oil horizons are in the basal beds of the Permian. The northern part of the Pool is an elongated dome, but the structure of the producing horizon is not clearly understood. There are indications that it is considerably more complicated than that of the overlying formations.

P. A. S.

144. Characteristics which Determine the Value of Oil Zones. G. O. Suman. *Oil Weekly*, 1930, **59** (13), 39–46.

Factors relating to sands from the point of view of their probable oil saturation are discussed. Attention is given to their designation, porosity, and gas and water indications. The author groups: (1) oil sands of gravity 30° B6, (2) those averaging from about 30° to 50°, and (3) above 50° B6. Tests made with various liquids on these sands are described.

Porosity is an important characteristic, in that it determines the quantity of oil that can be held in a sand and the effective drainage area of the oil toward the well. The greater the amount of such material as clay in a sand, the less favourably it is regarded owing to

the filling of the voids in the sand, which might otherwise be occupied by oil or gas. Fine and coarse sands are considered in this connection.

The possible presence of gas and water in oil sands must be borne in mind. In cable-tool holes gas may show in the bailings by bubbles and foaming, such indications being important as the deposit may be a *gas* sand and not one containing oil. Barren sands are not common in oil zones and should be suspected as water carriers until proved otherwise. A thorough knowledge of the general underground water conditions is therefore essential in dealing with such deposits, as water sands of high head and large volume will prevent any successful test of an oil zone if included in the area to be tested. G. S. S.

145. Evolution of Hydrogen Sulphide in the Bay of Krasnovodsk. D. Zavriev. *Azerbaijan Oil Ind.*, 1930, No. 10, 58–63.

Evolution of hydrogen sulphide is due to anærobic bacteria (especially "Mikrospira"). The organisms reduce sulphates into sulphides. Sulphides in turn are oxidised into sulphates by "Beggiatoa," and kindred micro-organisms. The sheltered position of the bay favours formation of undisturbed water layers and bottom stratum of silt. This and seasonal changes of temperature tend to make the process periodic. Examination of the silt confirms this view. Chemical and physiological investigation of the conditions at the bottom of the bay might throw some light on the origin of petroleum. P. A.

146. Bituminous Sandstone near Vernal, Utah. E. M. Spieker. *U.S. Geol. Surv. Bull.*, 822c, 1930, 77–98.

Four miles S.W. of Vernal, Utah, is a ridge of low hills, Asphalt Ridge, containing beds of bituminous sandstone. The rock is in the Uinta Formation of Eocene age, and is separated from the Mesaverde Formation of the Upper Cretaceous by an angular unconformity. The rock for the most part is a loose sand apparently cemented by the bitumen. The outcrop is 11¼ miles long and the average thickness is 72 ft., the exposures ranging from 16 to 190 ft. The saturation varies, the saturated portions being frequently parted by narrow clay bands and thin beds of conglomerate.

The rock gave 9% to 15% soluble in CS_2. The sulphur content (Carius method) was 0·09% and the fixed carbon 7·1%, both on the basis of the original rock. The estimated reserves, assuming a mining limit of 1¼ miles from the outcrop, are 1,970,000,000 tons. Mining will be necessary, as quarrying will touch a small part only. The rock makes a good paving material, and may possibly be a useful source of bitumen for hydrogenation into lighter fractions. G. R.

Field Technology.

147. Predetermination of Soil Corrosion. J. F. Putnam. *Oil and Gas J.*, 1930, 29 (26), 158, 187.

A simple ammeter voltmeter test has been devised which may be used to predetermine the corrosivity of soil in which iron is buried.

The sample for test is compressed in a bakelite box between two steel end pieces which have been previously cleaned with emery cloth.

The dimensions of the box are such that the final result is a soil cube of unit dimensions.

The resistivity of the cube is measured between the two end plates by means of an alternating current bridge (R_{sp}).

The end plates are then connected through meters and a potentiometer to a battery, and the potentiometer adjusted till the voltmeter shows a causation potential, E_c, of 1·4 volts. As this current starts to flow decomposition products are built up on the steel end plates, setting up a counter potential which reaches the decomposition potential E_d in from 5 to 10 min.

The current value then is $I = \frac{E_c - E_d}{R_{sp}}$

This current, read in milliamperes, is the corrosivity of the soil if causation potential E_c is 1·4 volts. The adoption of 1·4 volts as the causation potential was the result of an extended investigation of some forty-eight soils.

In general the method gives a rather close correlation to the corrosion experience on buried pipe-lines, except in extreme cases where the electrical resistance of the soil is very low and steady long line currents of considerable magnitude may be set up which materially affect corrosion. By long currents is meant currents of electricity which flow for several feet before leaking off into the soil, for which no method has been devised to predetermine either their direction or amount.

W. L.

148. Purification of Sea Water for Steam Boilers. I. Shick and G. Torosian. *Azerbaijan Oil Ind.*, 1930, No. 10, 104–108.

Absence of adequate supply of cheap, fresh water on the Apsheron Peninsula makes it imperative to use sea water for industrial installations of the Azneft.

Analyses of water from Baku Bay gave the following results:—Ca, 0·0355; Mg, 0·07075; Na, 0·2946; Cl_2, 0·5176; SO_4, 0·2933; Bicarbonates (as CO_3), 0·0143; Carbonates (as CO_3), 0·00044 g./100 c.c.

According to methods of Bunte and Aitner, 0·317 g. Na_2CO_3 per 100 c.c. of water will precipitate 95% of Ca. However, it was found sufficient to use only 0·075% of Na_2CO_3 in order to make the water usable. The above quantity of soda will remove half of the Ca as precipitate. By maintaining the water in boilers at 10° Bé, the remaining Ca will be precipitated as $CaCO_3$, and not as harmful $CaSO_4$ (scale). The boilers should be cleaned periodically. The conclusions were not verified on an industrial scale.

P. A.

149. Use of Superheated Steam in Drilling. V. W. Smith. *Oil and Gas. J.*, 1930, **29** (31), 83, 156, 159, 160, 162, 165, 167.

Although the cost of equipment has been mounting much faster than the return obtained from wells, little attempt has been made to build it more efficiently. In few instances has any attempt been made to reduce the power required or the weight per unit of work produced.

The author suggests that the principal reason for the slow introduction of efficiency into steam power plant equipment for drilling has been twofold—(1) most requirements in power plant equipment have been looked upon as fuel savers and the driller has no fuel cost, and (2) until recently the size of the power plant was too small for most

requirements to justify their cost. It is probable, however, that the increased capacity of the locomotive obtained by the use of superheaters more than justifies their use even though the primary objective was a saving in fuel and water.

The author discusses at some length what superheated steam is and how steam is superheated, and gives ten advantages that are offered by its use—(1) Reduction of steam consumption. (2) Reduced radiation losses (3) Reduced steam pressure. (4) Increased pressure of fluid pumped to the well. (5) Increased capacity, speed and flexibility of drilling rig. (6) Better operating conditions mean increased life of pumps, engines and boilers. (7) Decreased weight of boilers. (8) Decreased amount of water required for boilers. (9) Reduced fuel consumption. (10) Increased boiler efficiency.

The results of numerous tests are given and various sketches are added to assist the explanation. L. W. C.

150. Cleaning Sour Crude Storage Tanks. R. B. Roaper. *Oil and Gas J.*, 1931, **29** (33), 34, 97.

The number of fatalities resulting from the handling of sour crude, particularly in the cleaning of tanks, renders essential adequate supervision and organisation of such operations.

The most important step in tank cleaning is the blanking off of all lines connected with the tank, excepting the steam lines. Several deaths have resulted from the leaking of sour distillates containing hydrogen sulphide into the tank while cleaning. The manhole plate is removed and the tank filled with water from a high-pressure hose to float out as much as possible of the lighter sediment in the tank. A portable pump removes the water and oil from the tank, which is then steamed for 36–72 hours. Two doors are cut out of the bottom ring of the tank, steaming being still continued as an extra fire hazard precaution. The steam is cut off and the tank allowed to cool. When cool, a sample of the gas in the tank is taken in a pipette and tested for hydrogen sulphide (reduction of colour of iodine-starch solution) and for combustible vapours. When the sample shows complete absence of hydrogen sulphide and less than 0·2% of combustible gas (as pentane), the tank is passed as safe for cleaning. Precautions against the liberation of gases from the stirring of the residue are taken by insisting on the use of gas masks by the cleaning gang, not more than three of whom are allowed in the tank at one time. Each man is also provided with thigh boots. Pits are dug at the side of the tank next to the doors cut out and as much as possible of the b.s. filled into the pits by means of wooden drags. The remainder is removed by wheelbarrows and slop carts. The b.s. is then pumped to the boiler-house pit where it is burnt in Duncan sludge burners. Six men equipped with gas masks then scrape and sand blast the tank. The tools used are of non-sparking metal. The door sheets are then replaced, if necessary painting the inside of the tanks with rubshell or aluminium paint to prevent the deposition of the pyrophoric iron sulphide, the atmospheric oxidation of which is responsible for so many explosions. Any iron sulphide present should be removed with a high-pressure hose, as it will not oxidise in a damp atmosphere.

Every man should be thoroughly instructed in the Shaffer method of artificial respiration. C. L. G.

151. Aluminium Foil Used for Tank Roofing. H. F. Perkins. *Oil and Gas J.*, 1930, **29** (28), 31.

Aluminium has proved very resistant to the ravages of hydrogen sulphide, organic sulphur compounds and atmospheric corrosion.

Aluminium foil 0·005 in. thick, rolled from chemically pure aluminium, is most suitable for use, being equivalent to 1 in. of good insulating material at probably one-tenth the weight and half the cost.

Asphalt may be used as the adhesive, but disintegration of the adhesive may cause the foil to become loosened at the edges. Locked seams can now be made by machine, ensuring mechanical continuity, vapour and moisture proofness. A. R. S.

152. Tubing Improves Producing Conditions. L. G. E. Bignell. *Oil and Gas J.*, 1930, **29** (29), 33, 119, 120.

As the percentage of water increased with the wells on the Oklahoma City Field it was found advantageous to tube them. By this method the percentage of water to oil was reduced and in some instances, where the wells had gone completely to water, the running of tubing restored them as oil wells.

The wells vary in depth from 2,500 to over 6,000 ft., and in size from 2½ in diameter to 4 in. diameter with some strings of the tapered type.

External upset tubing and bottom hole chokes are used on the lower end of the string. Tubing wells have shown in every case a reduction in the gas-oil ratio.

The Reda pump has been tried with satisfactory results and a description of this pump is given.

The problem of sand cutting is a serious one and is dealt with in some detail, and a new type head to overcome this difficulty is explained.

The flow tanks, in which the stream is given a whirling motion, tending to separate oil, gas and sand, and the method of dealing with these constituents are described. L. W. C.

153. Texas Deep Wells Successfully Tubed. L. G. E. Bignell. *Oil and Gas J.*, 1930, **29** (29), 32, 121.

This article deals with the tubing of the 8000 ft. wells in the Big Lake Pool, Texas.

The first well to be tubed was 8603 ft. deep, cased to 7950 ft. with a string of 5$\frac{1}{2}$ in. 20 lb. casing. The time taken to run and set this string was 6 days.

The tubing crew commenced its work by lubricating in about 62 ft. of perforated pipe. The perforations are $\frac{3}{4}$ in. drilled holes. To get this into the well under the flowing pressures necessitated the rigging up of a 60 ft. lubricator over the well mouth. No difficulty was experienced.

A cast-iron plug was run above the upper end of the perforated pipe and this was held to its seat at the bottom by the pressure of the flowing oil and gas from the well. The addition of further lengths of tubing enabled the string to be lowered against the upflow of oil and gas.

Two control heads were used at the top of the well to facilitate running the 8465 ft. of 3 in. tubing.

After the string had been lowered the cast-iron plug had to be removed. Water was run into the 3 in. tubing, and when it was partly filled valves were opened between the casing head and the top of the string of tubing so that the well pressure was directed into the tubing on top of the column of water. When the pressure became enough the plug at the bottom of the well was forced down inside the perforated string and the well was then ready to produce through the tubing.

Before this well was tubed it made approximately 2500 brls. of oil per day with from 24,000,000 to 30,000,000 cu. ft. of gas, but as the tubing was lowered both the oil and gas flows were reduced, and on completion of the tubing job the well only made about 1700 brls. of oil per day with gas-oil ratio of about 9000 cu. ft.

Other wells have been, and others are in process of being, tubed, and the question of tubing of these deep wells appears to be feasible from the data available at present. These wells have not been producing long enough to give any really definite data as to what may be ultimately expected of them in the way of reduced gas-oil ratio, but the results show that the amount of oil produced through the tubing will be materially reduced and the gas flow per barrel has been cut about 2000 cu. ft. L. W. C.

154. Effect of Sulphide on Drilling Lines. L. W. Vollmer and B. B. Wescott. *Oil and Gas J.*, 1930, **29** (22), 64.

In wells where the amount of hydrogen sulphide encountered is large, embrittlement of cable on drilling tools is encountered. This embrittlement is similar to that due to nascent hydrogen.

Nascent hydrogen can be produced by the interaction of hydrogen sulphide and steel according to the equation

$$H_2S + Fe = FeS + 2H$$

Occlusion of this freshly generated hydrogen produces embrittlement in the lines.

The embrittlement of sulphide treated wires was measured by fastening one end of the wire in a horizontal chuck, the other in a vertical fixed clamp and measuring the number of revolutions required to cause failure. The conclusions arrived at are that this method is sensitive in measuring embrittlement in steel wire. Plain carbon steels become rapidly embrittled when subjected to the action of hydrogen sulphide in presence of moisture.

The embrittlement is caused by the absorption of nascent hydrogen produced by interaction of hydrogen sulphide and metal.

In cold worked steels the embrittlement is proportional to the degree of cold working and not the carbon content.

In dead soft annealed plain carbon steels, the embrittlement is inversely proportional to the carbon content.

Absorption of atomic hydrogen occurs mainly in the purest ferrite.

Steel wire embrittled by hydrogen sulphide recovers nearly all its ductility by standing for a time or gentle baking for a few hours.

The life of uncoated steel drilling lines in a sulphide field may be substantially increased by systematic periods of rest or by heating to 400° F. for 3 hours.

Galvanising and lead or copper coatings form efficient protective coatings, but tin is worthless. A. R. S.

155. Study of Drilling Speeds Used in Russia. P. Galiakav. *Petr. Engr.*, 1930, 2 (3), 77, 78, 80, 83.

The study of speed of drilling has attracted the attentions of various operators, and in the Grozny oilfields a fairly comprehensive study has been carried out, with a view to increasing the efficiency of drilling operations by a correct interpretation of the major factors involved. These factors are closely related to the factors affecting the cutting of metals.

The speed of actual drilling depends upon (a) the cutting speed and (b) the rate of feed. Cable and rotary systems each have their particular problems.

All factors affecting the speed of cutting can be divided into four groups—(1) Rig equipment, (2) Tools, (3) Well conditions and (4) Labour, organisation, etc. These four groups are dealt with individually both for cable and for rotary drilling.

The problem of rate of feed is a difficult one, dependent as it is on several very complicated factors. Graphs and tables are given.

L. W. C.

156. Delayed System of Drilling in the Water Flooding of Oil Sands. P. D. Torrey. *Petr. Eng.*, 1930, 2 (3), 111, 112, 114, 116.

The delayed system of drilling consists of delaying the drilling of the oil well on the "five spot square" until the sand body within the square is completely saturated with fluid.

When the input wells at the corners of the square will take no more water the well in the centre of the square is drilled for production. It was originally planned as a means of controlling a known and troublesome sand condition, but it has since been applied to almost all known conditions.

It has been accepted, not only because of its efficiency, but also because of the ease with which delayed floods may be operated when proration schedules of production are required. The system has provided a means of control that reduces operating expenses in partial proportion to the curtailed yield.

The results obtained, so far, show that a considerable increase in recovery may be anticipated both in the uniform and in the variable sand bodies.

L. W. C.

157. Great Savings in Operations at Van. L. E. Bredberg. *Oil and Gas J.*, 1931, 29 (33), 31, 115, 116.

Considerable economies have been effected by unitisation of operations in the Van Field, Van Zandt County (East Central Texas). The producing areas of several companies are operated by one company. The gas is run to their 14 unit gasoline plant in Nacogdoches County School Survey, and the crude (restricted) production of 27,500 brls. per day is run direct through a 10 in. pipe-line to Smiths Bluff on the Gulf Coast. The casinghead gasoline is also shipped by tank-car to this refinery.

Unitised operation has prevented duplication of production staff, field and pumping equipment, water systems, tank farms, etc., probably saving the five owning companies millions of dollars as well as effecting great reduction in oil and gas wastage.

The Van Field has been in operation under this scheme for thirteen months and is the first major field under unit operation. Its example will probably be followed by all newly discovered fields under joint ownership.

Descriptions of the compressing, fractionating and pumping equipment are given. C. L. G.

158. Gas in Relation to Oil Production. B. J. Ellis. *J. Inst. Petr. Techn.*, 1931, **17**, 2–29.

This paper consists mainly of relating the theories expounded by S. C. Herold in his work entitled "Analytical Principles of the Production of Oil, Gas and Water from Wells," to the conditions existing on the fields.

The problem of production is not merely one of mechanics. It is complicated by economics, and resolves itself into a compromise between the highest possible mechanical efficiency and the practical economic requirements.

The doubts which exist as to the absolute truth of Herold's theories, and the varying conditions which exist in the reservoirs, necessitate the adoption of some practical criteria towards which the oil engineer should aim. The author has formulated those necessary in the production of gas with oil.

Examining the composition of natural gas found in association with oil it becomes apparent that certain of the constituents will exist always as gases, others will exist as liquids under certain conditions, and the remainder will always exist as liquids. From these conditions some idea of the energy available can be obtained, and the author shows how this potential energy can be calculated.

Reservoirs are divided into three categories: 1, Reservoirs under hydraulic control; 2, Reservoirs under volumetric control; and 3, Reservoirs under capillary control. These are examined in some detail and the results obtained are then applied to practical conditions.

In the first case, the static head of the water remains constant, as the water is replenished continuously, and provided the friction head is also constant the rate of production will continue unchanged.

In the case of volumetric control the production of all the mobile oil in the reservoir will occur if the sum of the water and gas energy is sufficient to lift the total volume of oil to the surface. The rate of production will decrease continually as no water will be fed in at the outcrop.

In dealing with the question of capillary control the theory that production is governed entirely by the laws affecting the movement of liquids and gases in capillary tubes, is not entirely agreed with, but as no better theory has been propounded it has been accepted for the purposes of this paper.

Detailed study of the movement of oil and gas through the interstices of the sand is given, and from this study certain deductions, concerning the area over which each well exerts an influence, are drawn. Where the circles of influences interfere, the vertical plane through their points of interference is a neutral plane and no oil on one side of this plane can be produced at the well on the other side. This is used by the author for his most original theory, which he calls "Centripetal Production."

Repressuring and gas drive are dealt with both from the theoretical and practical view-point, and the paper concludes with an examination of centripetal production.

Using the theory of circles of influence expounded by Herold, the author suggests that by surrounding the output well by a sufficient number of input wells there is a possibility of doubling or even trebling the total ultimate yield, without departing in any way from the normal field methods. The input wells should, theoretically, be drilled so that their casings touch, but this is, of course, impracticable, and the number of wells would be decided by the percentage interferance of influence circles for the particular well spacing adopted.

The input wells could be small in bore and cheaply drilled, and this procedure may well result in higher ultimate yields being obtained.

L. W. C.

159. Theory of Curves of Draining of Oil Reservoirs. L. Leibenson. *Azerbaijan Oil Ind.*, 1930, No. 10, 64–66.

Curves of draining of natural oil reservoirs resemble the curves of draining of reservoirs, which are broad of base, narrowing towards the top. The concavity of such curves is towards positive ordinates. P. A.

160. Dynamometer an Aid in Correcting Well Pumping Troubles. H. Decker. *Petr. Eng.*, 1930, 2 (3), 65, 66, 68.

Although wells are being drilled to greater depths and pumping problems are becoming much more difficult, the author shows how the use of the Dynamometer has assisted in correcting pumping troubles as well as in furnishing helpful information regarding well operation.

Pressure charts were taken at various wells and by changing the size of the cup leathers and varying the counterbalance load a more efficient operation and a greater volume of fluid was obtained.

The effect that speed and length of stroke have on plunger travel and pump efficiency are illustrated and the result obtained by increasing the travel of the polished rod by 2 ft. per minute only is of interest. In addition to increased production the number of breakages and shut-downs was considerably reduced by this more efficient operation.

By an intelligent application of the data obtained by means of the dynamometer, an analysis of pumping troubles in a well can be made and from this analysis corrective measures can be suggested.

A variety of charts and diagrams are given and these show clearly that with correct counterbalancing more ideal conditions can be realised.

L. W. C.

161. Mechanics of Oil Well Pumping. H. E. Dralle. *Oil Weekly*, 1930, 59 (11), 24, 82.

In order to attain economic well pumping, a thorough study of both the surface and subsurface equipment must be made.

In the past, the study of oil well pumping has been confined mainly to the performance of pumps and sucker rods, and very little attention has been paid to the stresses set up in the surface equipment. The author points out that the stressing of the equipment is directly dependent upon the type of prime mover used, and upon the type, location, and amount of counterbalance.

The equations, test data, and results given help to simplify the theoretical discussion of this subject.

Twelve definite conclusions are arrived at by the author, and these should prove of great interest to those concerned with the study of oil well pumping. Briefly they are—(1) For maximum beneficial effect for protection of the surface equipment interposed between the prime mover and the crank a rotating type counterbalance is preferable. (2) Motors are preferable to gas engines. (3) Single cylinder engines superimpose high peak stresses on the surface equipment. (4) Analytical method of computing weight of rods and column of fluid and then taking a definite percentage of this remains the simplest method of counterbalancing gas-engine driven wells. (5) With band wheel drive the torques on the low-speed shaft showed little variation with counterbalance; motor torques showed very definite variations. (6) With multiple reduction gears the best counterbalances would indicate the best behaviour. (7) On electrically operated wells, the best criterion of the counterbalance is equal current peaks to minimise motor heating. (8) On the gear drives the counterbalance giving the best motor heating condition will give the best operating conditions on all surface equipment. (9) Rod stress is an insensitive indication of counterbalance. (10) Motor operated wells can be satisfactorily and easily counterbalanced by using a wattmeter, ammeter, or tachometer of the graphic type. (11) Extreme overloads on motors make them operate inefficiently. (12) The difference in time on the up and down strokes is not a criterion of counterbalancing.

L. W. C.

162. Constant Velocity Cycle in Slow Speed Pumping. C. L. Moore. *Petr. Eng.*, 1930, 2 (3), 101, 103, 104, 106, 108.

This paper deals in some detail with the results obtained from a series of investigations to determine the effect of slow-speed pumping of small wells. Slow-speed pumping is proving itself to be the cheapest and easiest method of lowering lifting costs on the small wells so that a profit can be made even with the low prevailing prices of crude. Speeds have been reduced in many cases to 5 and 10 strokes a minute, and in some cases individual wells are operating at 3 strokes per minute.

The experiments undertaken were based on Uren's theory of critical fluid levels in pumping wells coupled with the work of Beecher and Parkhurst on the solubility of gas in oil. A short discussion of the standard plunger pump is given.

Attention was particularly directed to the pressure losses and three conclusions were arrived at.

1. Pressure losses could be controlled within certain limits by the design of the standing valve.

2. Pressure losses increased rapidly with increases in the viscosity of the oil, which was beyond control.

3. Pressure losses increased directly as the velocity of the fluid through the standing valve, which is controlled by the rate of pump displacement or velocity of the plunger.

The third conclusion, involving plunger velocities, appeared to offer a solution and the constant velocity cycle pumping unit described is the result of these investigations.

The various pumping units are discussed and the author concludes with three major advantages of slow-speed pumping gleaned from practical experience of this work during the past five years.

1. Volumetric efficiencies of 90% or more are usually secured. This efficiency was obtained with a 2" pump operating only seven tenths of a stroke per minute (36") from a depth of 2200 ft. A constant velocity cycle was used.

2. With the use of constant velocity cycles, production increases are general.

3. Substantial reductions in lifting costs were always secured.

L. W. C.

163. THEORY OF CENTRIFUGAL PUMPS FOR VISCOUS FLUIDS. V. Baklanov. *Azerbaijan Oil Ind.*, 1930, No. 10, 120–124.

From theoretical considerations the following conclusions were reached—1. Pumps for viscous fluids require more blades than hydraulic pumps. 2. For great pressures more blades are required. 3. Rapid pumps can work with fewer number of blades. 4. Viscosities being equal, light fluids need less blades. P. A,

164. BACK PRESSURE JOINT. J. D. Lemex. U.S.P. 1,785,271, 16.12.30. Appl., 5.6.28.

This back pressure joint consists of a hollow body with the bore smaller than the drill-pipe to which it is attached and the lower end of which terminates in a valve seat. Passing through this valve seat is a valve guide which is supported by the main body and through the guide is fitted a spring valve. The guide is smaller in diameter than the valve area, to prevent uninterrupted flow of fluid through the joint.

L. W. C.

165. REAMER. O. Duda. U.S.P. 1,786,484, 30.12.30. Appl., 2.4.27.

This invention consists of a steel body equipped with a cutter carried by a pin which is permitted to slide in guides provided. A plunger operated by a spring engages the cutter and locks it in any predetermined position. L. W. C.

166. CASING RIPPER. R. Althunsen. U.S.P. 1,786,590, 30.12.30. Appl. 4.2.29.

The casing ripper consists of a steel body slotted to allow an eccentrically pivoted cutter to protrude and fitted with a spring to force the cutter into cutting position. Into the end of the slot a bolt projects to draw back the cutter and a latch is provided to hold the bolt in the withdrawn position. L. W C.

167. OIL WELL TESTING DEVICE. E. H. Cox. U.S.P. 1,786,897, 30.12.30. Appl. 4.2.27.

The device consists of a tubular member adapted for attachment to the lower end of a drilling string. A sliding sleeve encircles the tube together with a stopper and packer which are secured to the upper end of the sleeve. Both the sleeve and the tube are perforated so arranged as to register when the sleeve is at the upper limit of its sliding movement. A means is supplied for holding the sleeve in position in which the perforations are out of register. L. W. C.

168. Oil Well Sealing Device. M. McMahan. E.P., 338,692, 24.10.29.

The device consists of a seal, screwed to the top of the well casing, to force the cement, introduced into the casing, between the casing and the well walls. A shoe, held by locking plugs to the body screwed on to the casing, carries an expansible packing member, which is forced against the inside of the body by drawing the shoe upwards by means of a threaded eyebolt passing through the top of the body. Ports below the packing member allow fluid to be pumped into the casing to force the cement between the latter and the well walls. This fluid also exerts an upward pressure on the shoe member, so further compressing the packing member and increasing the effectiveness of the seal. J. M. F.

169. Apparatus for Measuring the Inclination of Boreholes. *Sun Oil Company.* E.P. 339,098, 21.11.29.

The apparatus consists of a water-tight casing in which a pendulum is supported for universal movement from the upper end, and a gyroscope is suitably mounted near the lower end of this casing. A film is carried by two spools, so that it passes across the casing between the pendulum and the gyroscope, suitably driven by an electric motor. Lamps carried by the pendulum, the gyroscope, and the brackets holding the spools provide spots of light shining on to the film. The lamps are provided with masks with various shaped apertures, so that their images can be readily distinguished. From these images on the film the orientation and deviation of the casing from the vertical can be determined. The wires operating the lamps are carried by the lowering cable. J. M. F.

Crude Oil.

170. Cold Settling of Surakhani Crude. A. Velikovski and S. Nifontova. *Azerbaijan Oil Ind.*, 1930, No. 10, 109–111.

Surakhani crude, diluted or not with naphtha, and also masut diluted with crude, were used for the experiments. The materials were subjected to natural cold, and crystals of petrolatum over 2 mm. in diameter were collected on a coarse cotton filter.

As an example, a mixture of 40% of Surakhani masut and 60% of naptha cooled to −24° C., yielded 27% of petrolatum. The deparaffinised product was distilled under an absolute pressure of 10 mm. mercury with the following results—Solar oils, up to 184° C., 28·2% wt. Pour point below −20° C.; light spindle oil, 184°–212° C., 11·2% wt. Pour point below −19° C.; spindle oil, 212°–238° C., 11·3% wt. Pour point below −7·5° C.; machine oil, 238°–272° C., 9·0% wt. Pour point below −8° C.; bright stock, residue, 40·2% wt. Pour point below 0° C. Experiments in a large laboratory and preferably on a semi-industrial scale are contemplated. P. A.

171. Electrical Dehydration Keeps Pace With Progress in California. W. A. Sawdon. *Petr. Eng.*, 1930, 2 (3), 57, 58, 60.

The most satisfactory type of dehydrator of crude oil emulsions depends upon the properties of the particular emulsion in question. Electrical dehydration is helping to solve the problem owing to flexibility in handling different types of emulsions. Improvements in design have been made to obtain better utilisation of the electric field, while a higher voltage is obtained by the use of two transformers and two separately insulated connections through the shell to the electrodes. The electrical dehydrator consists of a vapour-tight shell in which is placed a live and a grounded electrode, the former being maintained at a potential of up to 11,000 volts (A.C.). The cut oil is heated to a predetermined temperature and pumped into the dehydrator. The effect of the electric field is to charge the particles of water, which act as series condensers, rather than conductors, the high voltage then disrupting the oil film enveloping the drops. Adjacent particles of water then coalesce, progressively forming larger drops, until they are large enough to settle out. The water is then drawn off from the bottom and oil from the top. An automatic circuit breaker (which closes the circuit only when the oil level is above the top of the dehydrator) eliminates the danger of ignition of the oil vapour with the air present in the tank.

Agitation is frequently resorted to in order to bring fresh emulsion between the electrodes, and to eliminate short circuits by raising the electrode about ¼ in.

The cost of dehydration is recovered by the increase in gravity and, therefore, higher value of the treated crude oil, while operating conditions are rendered less difficult. C. L. G.

172. Distillation of Crude Oil. F. Tinker. U.S.P. 1,779,222, 21.10.30. Appl., 9.2.27.

In a topping plant consisting of a fire-heated pipe-still, a mixing chamber, a separating chamber, a plurality of fractionating towers and fractional condensers, and a condenser for light distillates, hot residuum from the separating chamber is taken through a heat exchanger in which crude oil before entering the pipe-still is subjected to a preliminary heating. Hot refluxed oil from one or more of the condensers is also passed through the heat exchanger and caused to heat cold crude oil which is passed next through a coil or pipes inserted in the fractional condensers for further heating and is finally delivered to the mixing chamber, where it mixes with the hot oil or vapour from the still. Crude oil is thus used for cooling the contents of effluents from the condensers, and also for condensing vapours, and is thereby itself heated to a temperature at which it can advantageously be mixed with the main stream flowing through the distillation system.

This process may also be applied to a similar system in combination with a branch circuit containing an additional still and superheater, condensate from a fractional condenser being supplied to the still by a pump, and the hot vapour from the superheater being delivered to the mixing chamber. A similar branch system may be used for cracking oil under pressure in the liquid phase. H. E. T.

173. Oil Still. D. S. Jacobus, Assr. to Babcock and Wilcox Co. U.S.P. 1,779,434, 28.10.30. Appl., 19.1.25.

In this invention a bank of tubes connected to an upper and a lower drum extends across the furnace chamber at an angle of 45°.

A bridge wall is provided in the chamber between the hot gas inlet and the waste gas exit extending from the floor of the chamber to immediately below the bank of tubes, thus defining a path for the heating gases and causing them to pass first upwardly across the upper ends of the tubes and then downwards across the rear ends to the waste flue. Through this bridge wall there is a duct connecting the waste flue with the front or hottest portion of the furnace chamber. By means of an air or waste gas injector a certain proportion of the cool waste gas is recirculated and utilised for diluting the hot incoming gases so as to prevent injury to the tubes and at the same time to allow the fuel to be burned at its greatest efficiency. W. L.

174. Distilling Liquids. J. Delattre-Seguy, Assr. to Universal Oil Products Co. U.S.P. 1,782,810, 25.11.30. Appl., 28.6.26.

The oil is passed through the heating chamber by causing it to flow down an inclined wide shallow channel. This channel is mounted on adjustable standards so that its slope may be varied at will. Oil is fed at the upper end through a transversely extending perforated nozzle, and at the lower end is delivered into a wide funnel which is connected to the residue run off. The vapours pass out of the heating chamber through a vapour collector dome and may be condensed at once or further treated in the vapour phase.

Heat is supplied from an open trough, made of carborundum or brick, above the plate and inclined in the opposite direction to it.

The desired temperature is attained by allowing molten material, such as metal or glass, to pass through this trough at any desired rate.

Chemical reactions between the cracked vapours and the medium in the trough may be set up which materially refine the vapours before they leave the heating chamber. W. L.

175. Heat Transfer System. A. E. Nash and J. S. Alcorn, Assrs. to Alcorn Combustion Co., Philadelphia. U.S.P. 1,782,829, 25.11.30. Appl., 3.1.27.

The patent deals with the control of the amount of heat supplied to the tubes in a pipe-still, particularly that supplied by convection, by fitting shields or baffles to the lower rows of tubes.

The baffles may be of various forms and may have different methods of mounting. A baffle may be circular in cross section and of greater diameter than the tube round which it is placed, or it may have a slot along the top, that is, it may be circumferentially discontinuous, thus exposing the upper sector of the tube to direct contact with the flue gases. A baffle may on the other hand have a V or a semi-circular cross section, and the ends may be built into the furnace walls, or it may be suspended from the tube at the desired distance from it.

The baffles may be composed of silicon carbide or, preferably, of a metal or alloy, such as iron, steel, nichrome, or the chrome iron and nickel alloys.

The baffles become incandescent and supply radiant heat to the tubes which they protect from too great absorption of heat by convection.

H. B. T.

176. Distilling Apparatus and Method. E. O. Benjamin. U.S.P. 1,784,956, 16.12.30. Appl., 28.7.24.

The apparatus is designed for the fractional distillation and cracking of crude petroleum or "topped" crude. The oil is passed successively through a series of retorts maintained at successively higher temperatures and lower pressures. The vapours from each retort are fractionally condensed, the heavier condensate being fed into the next retort. A rapid circulation of the oil in each retort is maintained by the convection effect produced by a relatively cool down-flow path from the upper to the lower portion. W. H. T.

See also Abstract No. 312.

Natural Gas.

177. Gas Industry in the Azneft. G. Petrosian. *Azerbaijan Oil Ind.*, 1930, No. 10, 10–17.

The gas production in the Azneft cannot be regarded as satisfactory. Much can be done to rationalise production of gas and to make its use popular on petroleum installations.

Production of crude oil and gas in 1928/29 was as follows:—

	Tons.
Crude Oil	8,370,000
Gas	159,000

The gas was consumed as follows:—

	Tons.	Percentage of total fuel.
Petroleum Installations	5,800	1.5
Electric Plant	40,600	18.5
Domestic Use	94,500	88.3
Industry	17,000	48.5
Drilling Installations	2,200	22.5
Gas Industry Department	1,600	100.0
Others	1,100	2.7

It is desirable to introduce production of carbon black and to improve recovery of gasoline from gas.

The gasoline content of the gas in gal./cu. ft. is Baku, 0·5; Grozni, 17·0; Surakhani, 1·3; Bibi-Eibat, 0·5. The Surakhani gas has a carbon content of 0·56 to 0·68 lb./1000 cu. ft., and a CO_2 content of over 15%.

Extraction and utilisation of CO_2 is recommended. It is advocated to revise the 5 years plan for the gas industry, enlarging its scope and usefulness. P. A.

178. Interest Now Increasing in Russian Gas. *Oil and Gas J.*, 1930, 29 (30), 126.

Not until 7 years ago was the value of natural gas as a source of natural gasoline fully realised in Russia. The change in attitude is attributed largely to the replacement of the old method of bailing wells by modern closed systems, since 80% of the natural gas is obtained from flowing oil wells. Statistics are given and show a reduction in the percentage of crude produced by bailing from 37·8 to 3·3 during the period 1923 to 1929.

The first natural gasoline plant was erected in 1923 in Grosny, where the gases are considered to be the richest in the world as regards natural gasoline content, particularly in the new production areas. Yields of 12 to 15 gal./1000 cu. ft. of gas are obtained. The capacity of the natural gasoline plants in this field is about 180,000 brls. of gasoline per annum, but an increase to 1,350,000 brls. is contemplated in the next 5 years. In Baku the gasoline content of the gases is relatively low, *viz.*, about 1 gal. per 1,000 cu. ft., and the present equipment allows the extraction of about 55,000 brls. per annum. An increase in capacity to 180,000 brls. is expected under the five-year plan.

The principal area in which gas is produced without oil is South Daghestan. Production data for this area during the last 2 years are not available, but previously just over 1,000,000 cu. ft. of gas daily was obtained.

Residual gas in Russia is largely used by the petroleum industry, and also for industrial and domestic heating. Statistics giving production of gas in various fields are included and show that the total Russian production has increased from 448,186,000 cu. ft. in 1913 to 9,364,097,000 cu. ft. in 1928–9.

The Geological Commission is engaged in studying those areas in which shallow drillings have revealed the presence of natural gas.

R. A. E.

179. Natural Gasoline Situation Changing. C. O. Willson. *Oil and Gas J.*, 1930, **29** (30), 98, 99, 136, 137.

The large actual and potential production in the Kettleman Hills Field, California, of both high gravity crude oil and natural gas, is having a profound effect on the market value of casing-head gasoline and also on the specification demanded by buyers. The ratio of production of natural gasoline to crude oil is relatively high (1 : 2), so the field is of particular interest to natural gasoline manufacturers. It has been estimated that the field is capable of producing 400 million cu. ft. of gas daily for 75 years. There are six modern plants in the field, each having a rated capacity of 75 to 100 million cu. ft. of gas per day, operating at 450 lbs. pressure. The gasoline recovery is 1·2 gallons per 1000 cu. ft. of gas. Five plants at present operating on gas from eight wells are producing daily 500,000 gal., or about 25% of the total Californian production of natural gasoline. It is expected that exports of Californian natural gasoline will increase next year.

The finished natural gasoline is practically free of propane, contains about 3% of iso-butane, 22% of n. butane, and the remainder consists of pentane and hydrocarbons of higher boiling point. The vapour pressure is 22 lbs. (Reid). Due to absorption by the crude oil, the percentage of hydrocarbons heavier than hexane in the finished product is lower than usual for Southern Californian natural gasolines.

The recovery on distillation is 96% as compared with 90% for the bulk of the natural gasoline a year or so ago, and it is anticipated that the higher recovery will now be accepted by all manufacturers and buyers in California. The situation has also been affected by the recent instalment of stabilisers by many manufacturers in California.

The crude oil is unusually light in character, yielding 75 to 85% of straight-run gasoline, and a light residuum free of heavy ends. This

results in absorption of some of the gas by the crude in the wells and traps, and one company is stabilising the crude oil coming from the wells in order to reduce storage losses.

A programme of curtailment of production is in force at the moment, and the future effect of the field on production and market values will depend on whether or not this limitation is continued. R. A. E.

180. New Shipping Method for Exporting Helium. *Oil and Gas J.*, 1930, **29** (31), 105.

It is becoming realised that helium is absolutely necessary for the safe operation of airships. Transport of helium to countries outside America can be carried out in steel cylinders. Lieut.-Comdr. C. E. Rosendahl, of the Bureau of Aeronautics Dept. of the Navy, suggests the construction of transport which could be filled with helium under pressure. Once the demand is sufficiently great, there should be no difficulty in working out an economic method. An alternative is to fly foreign airships to America filled with hydrogen and there to reinflate with helium. There is no doubt, however, that successful commercial airships of the future will have to be helium filled. C. L. G.

181. Separation of Gaseous Mixtures. W. K. Lewis, Assr. to Standard Oil Development Co. of New Jersey. E.P. 338,719, 11.11.29.

A method is described for obtaining a gas rich in hydrogen from a less concentrated mixture composed of hydrogen and hydrocarbon gas. The mixture of gases is brought into contact with activated charcoal under a pressure of more than 20 atm. and at a temperature less than 100° F. until a substantial saturation occurs. The unadsorbed gas rich in hydrogen is withdrawn and a gas rich in hydrocarbons is passed into the body of saturated charcoal, thus displacing the unadsorbed gas held between the grains of charcoal. The pressure on the saturated charcoal is then reduced slowly and part of the gas evolved is recompressed and used to displace unadsorbed gas from the saturated charcoal in a following cycle, several bodies of charcoal being utilised alternately in cycles. W. S. C.

182. Catalytic Desulphurisation of Gases. J. Y. Johnson, Assr. to I. G. Farbenind A.-G. E.P. 340,016, 16.9.29.

Gases are desulphurised by heating in the presence of hydrogen with catalysts consisting of mixtures of the sulphides of the elements of groups 2 to 7 of the periodic system with metals of group 8 or with heavy metals of group 1 or their compounds. The hydrogen sulphide formed is then removed. The catalysts are deposited on carriers and are maintained at temperatures between 200° and 400° C. W. H. T.

Motor Fuels.

183. Revised Grading of Natural Gasoline. W. T. Ziegenham. *Oil and Gas J.*, 1930, **29** (32), 30, 102.

A new system of grading natural gasoline, based on vapour pressure and volatility, has been adopted by the Natural Gasoline Association of America. It is now considered that vapour locking tendencies are best indicated by the Reid vapour pressure and ease of starting, warming

up performance and accelerating ability by the A.S.T.M. distillation temperature at which 10, 35 and 60% are evaporated, respectively. It is stated that vapour pressure and volatility provide adequate information for blending natural gasoline, and it is considered that the new specification will enable the manufacturer of blends more easily to choose the grade of natural gasoline he requires for his purpose, and to be more certain of the quality of his final product. At the present stage of laboratory technique, specifications based on composition are considered unsatisfactory.

Natural gasoline is defined in the official specification as having the following properties: Vapour pressure, 10–34 lbs./sq.in.abs. at 100° F. (Reid); Evaporation at 140° F., 25–85%, at 275° F. not less than 90%; E.P., not higher than 375° F.; Corrosion, non-corrosive; Doctor, "sweet"; Colour, not less than +25 (Saybolt). In addition, natural gasoline is divided into 24 grades. There are six subdivisions according to Reid vapour pressure at 100° F., each having a range of 4 lbs. (maximum 14, 18, 22, 26, 30 and 34 lbs. respectively). Each subdivision is again classed according to evaporation at 140° F. in ranges of 15% (minimum 25, 40, 55 and 70% respectively). The individual grades are thus described as 14–25, 34–70, etc.

The A.S.T.M. distillation point of 140° F. applies to barometric pressures of 740 mm. For other barometric pressures, the temperatures shown in the following table must be used.

Pressure mm.	Corrected temperature ° F.	Pressure mm.	Corrected temperature ° F.
754	141	669	135
740	140	655	134
726	139	641	133
712	138	626	132
697	137	612	131
683	136	598	130

Additional corrections shall be applied at the rate of 1° for 14·5 mm.

The methods of test are generally those laid down by the A.S.T.M., but modifications are made in the distillation test. A Doctor test for general use for natural gasoline has also seen standardized. (See Abstract No. 291.) R. A. E.

184. Aircraft Diesel Engine Fuels. W. H. Graves. *Aut. Ind.*, 1930, **63**, 892–894.

This paper was read at the A.P.I. Meeting in Chicago in Nov., 1930, and the first part deals with the essential differences between Diesel and gasoline engines and the second section with the fuel handling system on a Diesel engine.

In the latter case, efficient injector pumps are essential to ensure good atomization, which, together with a high degree of turbulence, is necessary for complete burning of the fuel.

The type of pump used on the Packard-Diesel engine is illustrated.

Recent tests indicate that anti-knock Diesel fuels are advantageous as they allow shorter injection periods and thus give more power.

Oils with the following tests have proved satisfactory: Sp. Gr. at 60° F., 37–42° A.P.I.; F. pt. (open), 150–180° F.; Colour, White–1½ A.P.I.; Viscosity at 100° F., 32–35 Saybolt Univ.; Viscosity at 0° F.,

45–50 Saybolt Univ.; I.B.P., 348–430° F.; E.P., 490–600° F.; Pour Point, −30 to −80° F.; Ash, Acidity, Alkalinity, nil; Sulphur, up to 0·5%; Water and Sediment, up to 0·05%.

The essential features of a Diesel aircraft fuel are—(1) a distilled fuel; (2) a pour point and viscosity low enough to flow at low temperatures; (3) a flash point over 150° F.; (4) absence of acidity, alkalinity and grit C. C.

185. Results With Power Alcohol in Sweden. E. Hubendick. *Pet. Zeit*, 1930, 26 (50), *Motorenbetrieb*, 3–9.

A short review of previous work including that of Meyer and Wawrainiok is given.

In the present research experiments were carried out on ten different types of Swedish commercial benzines and mixtures made from 80% of benzine and 20% of alcohol or spirit. The strengths of the spirits varied from 0·5%, 99·5% to 97·5%, and the turbulence points of all the mixtures were found. The demixing of a fuel mixture occurs in two stages, first turbulence or opalescence is observed and finally a separation into two layers takes place. It was observed in Sweden that those mixtures which remained homogeneous at −30° contained a spirit of a strength not less than 99%; fuel mixtures containing no greater than 20–25% of alcohol were most useful.

Further experiments were performed utilising benzine and benzine-alcohol mixtures in varying proportions in fuel mixtures. Graphs are given showing the relation between the H.P. and the heat units/H.P. hour for the various blends, also of the relations between the % by weight of alcohol in the mixture and the heat units/H.P. hour.

The good qualities of "lättbensyl," *i.e.*, a mixture of 75% of benzine and 25% of technical alcohol, are described. It can be applied instead of benzine and causes no demixing. It is knock-free and increases the power of the engine and decreases the fuel consumption as compared with benzine; moreover it causes no corrosion or deterioration in the engine. W. S. C.

186. Refining of Motor Benzol by Means of Silica Gel. R. C. Geddes. *Gas World*, 1931, **94**, 17–20.

Crude benzol, direct from the stripping stills, is warmed to a temperature of 40° C., given two very light sulphuric acid treatments, (*a*) 0·1% to dry the benzol, and (*b*) 0·2% in a separate mixer to start polymerisation. After settling, the acidulated product passes through coke filters to the silica gel, contained in the percolator, entering at the bottom. The benzol passes upward through the charge of silica gel at a temperature of about 150° C., at a pressure sufficient to maintain it in the liquid phase. After this process, the heated product is given a light caustic soda wash and then passes on to a rectification still

Compared with sulphuric acid refining, increased yields to the extent of 13% are obtained by silica gel treatment, due to practically all the stable unsaturated compounds remaining in the finished product, only the unstable gum forming diolefines being removed by selective action of the silica gel.

When testing motor benzol refined by this process, the copper dish test is recommended in place of the acid wash test. W. A. W.

187. Treating Light Oils. G. Staub. *Petr. Eng.*, 1930, 2 (3), 161–164, 166.

The presence of sulphur in light oils, beyond a certain limit, is objectionable. The chief sulphur compounds which can be removed by treatment are—Free sulphur, hydrogen sulphide, organic sulphides, mercaptans, disulphides and thiophenes.

Hydrogen sulphide is found in practically all crude oils and distils over to a large extent as gas. In the presence of water it has weak acid properties and is highly corrosive to the distillation equipment. It is usually neutralized by the introduction of ammonia or caustic soda into the condenser coils, pipe-lines, etc.

Mercaptans react with sulphuric acid to form disulphides, or with doctor solution Na_2PbO_2 to form lead mercaptides. Both resulting compounds are soluble in the gasoline. Organic sulphides do not react with doctor solution, while the disulphides can be dissolved in strong sulphuric acid. Thiophenes are also soluble in sulphuric acid.

From the point of view of the refiner the most important sulphur compounds are hydrogen sulphide and the mercaptans. A gasoline is usually not considered satisfactorily refined unless it is negative to the doctor solution. For this reason, refining with caustic soda and doctor solution is generally found adequate for sweetening gasolines. The former (usually of 20–25° Bé. strength) removes the hydrogen disulphide, the resulting sodium sulphide being insoluble in oil and removed by water washing.

Mercaptans are not so easily removed with doctor solution since the lead mercaptides formed are soluble in the gasoline if present below a certain limit. The lower mercaptans give mercaptides less soluble in the gasoline than the higher ones, while the higher mercaptans react very slowly with doctor solution owing to their insolubility in water.

The higher mercaptides, soluble in the gasoline, are best treated by the addition of sulphur to the doctor solution, resulting in the formation of alkyl sulphides, soluble in the oil, but not reacting positive to the doctor test. Lead sulphide is then precipitated as a black sludge. Care must be taken that too much sulphur is not added, owing to its solubility in gasoline. The heavier mercaptans, not amenable to doctor treatment have to be removed with sulphuric acid.

Gasolines and kerosines with high natural sulphur contents have to be treated with sulphuric acid, the reactions involving sulphonation, addition, polymerisation, formation of alcohols, and general solvent action. The result of polymerisation is to raise the final boiling point of the product, so that acid treatment should be carried out before final distillation, especially in the case of cracked gasolines.

Heavy mercaptans can also be removed by cupric chloride treatment, resulting in the formation of alkyl disulphides, soluble in the oil. In this case no sulphur addition is necessary. Care must be taken, however owing to the corrosive nature of cupric chloride solution. Further reduction of sulphur can be effected by acid treatment, preferably 66° Be. sulphuric acid in two stages. C. L. G.

188. Ethylene as Possible Source of Liquid Fuel. G. T. Morgan, H. Ingleson, R. Taylor. *Petr. Times*, 1930, **24**, 1024.

Commercial ethylene under pressure of 70 atm. in the presence of a basic zinc chromite catalyst was passed through a copper-lined converter ½ in. internal diameter, heated to 450° in an oil bath. A light yellow oil with a greenish fluorescence was obtained. The average molecular weight of the crude product was 115, while the density at 15°=0·754 and 100 g. of oil absorbed 116 g. bromine. Five fractions were obtained by distillation.

			Bromine Number.
Up to 80° C.	.. 415°	0.685 ..	195
80–118° C.	..	0.731 ..	133
118–152° C.	..	0.756 ..	114
152–185° C.	..	0.775 ..	98
Above 185° C.	..	0.827 ..	—

Fractions 1 and 2 were water white, 3 and 4 were light yellow, and the residue was a dark brown fairly mobile liquid.

Engine tests showed that the H.U.C.R. of fraction No. 1 was 5·4 approx., and the 3 fractions boiling up to 185° C. had an H.U.C.R. of 4·8 approx. The remaining fraction was unsuitable as a fuel. The initial ignition temperature was 300° C. approx. for all fractions. All fractions were liquid and free from separation at –60° C. Attention is drawn to earlier work by W. I. Patiev. (*Ber.*, 1911, **44**, 2978–2987). H. G.

189. Propagation of Combustion in Carburetted Mixtures. R. Aubert and R. Duchêne. *Ann. des. Comb. Liquides*, 1930, **5**, 737–739.

In order to use higher compression ratios, the glass tube forming the explosion chamber is replaced by a metal cylinder, fitted with a rectangular glass window to enable the flame travel to be followed photographically through the whole length of the cylinder. The compression ratio is 5·9 and ignition is by spark at the end of the compression. A temperature of either 15° C. or 50° C. to 53° C. is maintained in the combustion chamber.

The flame speeds in mixtures of benzene, benzol, hexane, heptane, amylene and No. 1 petrol were measured in this apparatus at mixture strengths of 108 to 191 mm.³ per litre and at initial temperatures of 15° C. and 50° C., and the following results and conclusions were obtained:—

The nature of the flame front is always as previously indicated, and the influence of cylinder temperature on the speed of the flame is considerable. (See C.R., 1928, **186**, 200, **187**, 220.)

General inflammation occurs with Hexane at 15° after 0·009 sec., with hexane at 50° after 0·003, with heptane after 0·037 and with No. 1 petrol at 50° after 0·005 seconds. In these cases the first pale flame is succeeded by a very actinic flame, throughout both the unburned and partially burned gases. With hexane at ordinary temperatures the general inflammation of the charge occurs after the flame has completely traversed the explosion chamber.

With benzene and other fuels tried, this effect was not observed; sometimes a return flame was noticed but this followed the initial flame immediately it was reflected from the bottom of the chamber. At 50° F. this general inflammation occurs with hexane, heptane and

petrol, but before the flame has completed its first travel. This effect does not occur with aromatics or blends containing large proportions of these.

The authors think that "knocking" in engines is due to this general combustion, which is different from the detonation to which knocking in motors is often attributed.

Experiments have been carried out with the object of correlating knocking and photographic results, using a 4% solution of amylene in the same No. 1 Petrol on a single cylinder engine with Midgley bouncing-pin. The amylene-blend knocked noticeably less. The straight petrol inflames generally in the photographic apparatus while the amylene-blend burns progressively. E. B. E.

190. Low Boiling Hydrocarbon Oils. Standard Oil Devel. Co. E.P. 318,521, 3.9.29.

The process provides means for reducing the formation of gum in relatively low boiling hydrocarbons by the addition of pyrogallol in an amount not less than 5 g./gal. (*e.g.*, 0·1 g./gal.).

191. Low Boiling Hydrocarbon Oils. H. G. M. Fischer and C. E. Gustafson, Assrs. to Standard Oil Devel. Co. E.P. 319,362-3, 19.9.29.

Claims are made for a low boiling hydrocarbon oil normally tending to form gum and stabilised against gum formation by the addition to the oil of a small quantity (0·1% by vol.) of an aromatic hydrocarbon with condensed nuclei (anthracene). The inhibitor may also contain an aromatic compound with condensed nuclei having an alpha substituted group which is able to combine with acids (e.g. alpha-naphthol).

Both patents are practically identical, except that in the second the hydrocarbon oil may contain a small quantity of a metal carbonyl or organo-metallic compound in addition to the aromatic compound. W. H. T.

192. Motor Spirits. L. W. E. Townsend and A. Richmond. E.P. 338,581, 23.7.29.

An oil possessing anti-detonating properties is prepared by cracking shale oils by a restricted and controlled distillation in which the dehydrated tar is heated to 120° C. in the presence of a slow current of inert gas, or alternatively in steam. This oil is added to similar distillates of petroleum, shale or coal tar of higher boiling point and not generally suitable for direct use in internal combustion engines. W. S. C.

193. Treating Petroleum Oils Containing Sulphur. G. J. Ziser and J. H. Osmer, Assrs. to Standard Oil Co. U.S.P. 1,784,215, 9.12.30. Appl., 9.1.26.

The invention refers particularly to the treatment of gasoline and kerosine obtained from naphthenic crudes of high sulphur content. The method consists of treating the oil with an aqueous solution of a copper ammonium salt saturated with dissolved oxygen. W. H. T.

See also Abstracts Nos. 179, 242, 250, 291, 292, 309.

Kerosine.

194. Refining Petroleum and Like Hydrocarbons. W. W. Triggs. E.P. 338,482, 7.5.29.

Lamp oil distillate derived from an asphalt or mixed base petroleum crude is first treated with liquid SO_2 at a temperature below 32° F., preferably 17° F., in quantities just sufficient to extract the major portion of the undesired hydrocarbons, after which the partly refined distillate is separated from the surplus SO_2 and treated with SO_3 or fuming H_2SO_4 in quantities sufficient to complete the purification. The acid treatment is carried out at temperatures below 32° F. and in the presence of dissolved SO_2. These conditions bring about a selective treatment without the extraction or chemical decomposition of any of the desired lamp oil stock.

The method may be worked continuously and, though primarily adapted for lamp oil distillate, lubricating oil stocks, distillates suitable for the manufacture of liquid petrolatum, or cracked hydrocarbons, may also be refined by it. W. L.

195. Refining Mineral Oils. W. W. Groves, A.-G. für Chem. Ind. G.m.b.H. E.P. 338,579, 22.7.29.

The patent deals with a process for refining mineral oil in which the distillate obtained up to about 400° C. is freed from the benzine fraction, the residue being subjected to treatment with liquid SO_2. The distillate may be treated with the liquid SO_2 without the previous removal of the benzine. In either case, the (SO_2) insoluble portions, after removal of SO_2, are subjected to fractional distillation. W. H. T.

See also Abstracts Nos. 187, 193, 243.

Lubricants and Wax.

196. Chemical Structure of Lubricating Oils. G. H. B. Davis and E. N. McAllister. *Ind. Eng. Chem.*, 1930, **22**, 1326.

An examination of the data on paraffin and cycloparaffin hydrocarbons has indicated that the relation of molecular volume to molecular weight offers a means of estimating the relative proportions of these structures in petroleum products. The relationship is indicated by the equation:

$$N = 0{\cdot}358 \text{ mol. wt.} + 7{\cdot}7 - 0{\cdot}3 \text{ mol. vol.},$$

where N is the number of carbon atoms per molecule in naphthene rings. When the equation is applied to various naphthene hydrocarbons, the calculated number of carbon atoms in the rings agrees within one of the actual number. The method had been applied to Pennsylvania lubricating oils, in particular, to vacuum distillate fractions obtained from a refined heavy motor oil and to solvent extraction fractions obtained from these distillates. The conclusion is arrived at that Pennsylvania type oils are composed essentially of a grouping of paraffin chains with

naphthene rings and the number of carbon atoms in napthene rings in oils of this type can be calculated from the molecular weight and density. The necessary molecular weights were determined by a cryoscopic method using cyclohexane as a solvent. The empirical formula for each sample is calculated from the number of carbon atoms in naphthene rings by assuming an average of six carbon atoms per ring and a deficiency of two hydrogen atoms per ring. The empirical formula calculated in this manner corresponds closely to that calculated from carbon-hydrogen analyses. The viscosity index appears to be a function of the percentage of carbon atoms in the molecules that are in naphthene rings. R. W. L. C.

197. Proposed Change in Specifications for Motor Lubricants. *Oil and Gas J.*, 1931, **29** (34), 122, 127.

The Federal Specifications Board has submitted a proposed revision of specifications for Class D. lubricating oil which involves a system of grading the oils numerically from a consideration of their main physical properties. The oil must meet a certain number of basic requirements and is thereby credited with an arbitrary value of 100 points, further points being given for superiority in other directions. To be acceptable the oil must be credited with a total score of 115 points. This method of evaluation is applied to 6 grades of oil for use in internal combustion engines, except aircraft and Diesel engines, which have S.A.E. numbers between 20 and 70.

The basic requirements are viscosity limits at 130° F. to 210° F., minimum flash point, and maximum A.S.T.M. colour, pour point and carbon residue. The oils must also be composed of pure mineral oils only.

Computation of the credit assigned to any particular oil is made by multiplying the difference between the requirements of any test and the actual value found for the oil, by a definite unit.

Thus if the flash point specification for oil S.A.E. 20 grade is minimum 335° F., the oil scores 0·02 points for every ° F. which its flash point is over that figure. Similarly it scores 0·5 per unit for superiority in A.S.T.M. colour value, and 1 unit for each 5° F. which its pour point, and 10 points per 1% of carbon residue, is lower than the specification.

Credits are also given for flatness of viscosity curve.

The oil must also have a Sligh oxidation number not over 40 and a neutralisation number not over 0·30, while it must not strongly discolour copper strip when tested by method 530.31. Credits are also assigned for superiority in the first two of these tests. C. L. G.

198. Acid Treatment of Lubricating Distillates. H. M. Weir, W. F. Houghton and F. M. Majewski. *Ind. Eng. Chem.*, 1930, **22**, 1293. *Oil and Gas J.*, **29** (26), 122, 173.

A laboratory method has been developed for determining rapidly the best conditions for optimum colour and oil yield during the sulphuric acid treatment of lubricating oils. The method consists in the addition of a small measured volume of acid to 100 c.c. of oil contained in a centrifuge tube. The centrifuge tube is mounted in a shaking machine and temperature control is effected by placing the machine in a cabinet

which can be maintained at any desired temperature between 80° and 170° F. After treatment is completed by about 40 min. agitation, the contents of the tubes are centrifuged at 1500 r.p.m. for 1 hour. The chief criterion employed to determine the efficacy of the treatment is the reduction of colour produced. The colours of the treated oils were determined in a Duboscq colorimeter. For this purpose the oil to be examined was first diluted with a mixture of extraction naphtha and medicinal white oil to approximate in colour to a suitable oil which has been adopted as a standard. The standard was a filtered Manchester spindle oil of 2½+N.P.A. colour. The colours were determined on the sour oil, the oil saturated with anhydrous ammonia and a finished product after soda washing and brightening. The oil used was a Solar oil from Seminole crude of viscosity 129 sec. Saybolt at 130° F. For the purpose of investigation the ammonia colour of the oil was adopted for the measurements as this most nearly approximated to the colour of the works soda washed product. The Solar oil was treated with acid of 97·8 to 75·0% H_2SO_4 over a range of 0·05 to 9·5 c.c. of acid per 100 c.c. of oil. 93% H_2SO_4 showed a superiority over other strengths. Thus for a certain colour 1·3 g. of 93% strength acid are required. To obtain this same colour with 98, 90·7, 86 and 82·6% acid, 1·45, 1·70, 2·3 and 3·6 g. of H_2SO_4, respectively, are necessary. Colour removal is less a function of the weight of H_2SO_4 used than of the concentration of the acid and bears no relation to the oil loss incurred in treating. 93% acid is not the most efficient for colour removal in all cases. Fluxed bottoms, for example, are usually treated much more efficiently with 98% acid. As with the colour, the loss on acid treatment is chiefly influenced by the concentration of the acid. The effect of temperature of treatment is not very pronounced in the case of this Solar distillate. At or below 60° F. the high viscosity reduces the efficiency of contact, and at 140° F. or above oxidation darkens the oil. Between 80° and 115° F. the colour is not appreciably changed for any amount of treating up to 15 lb. of acid per 50 gal. brl. The treating activity of a weak acid cannot be increased to equal that of a strong acid by merely raising the temperature. The oil loss does not change with temperature.

The effect of various amounts and different concentrations of acid on gravity, viscosity and refractive index of the oil have also been determined, and here again concentration of the acid is a more important factor than the amount employed. The heat of acid-treating reaction has been determined, and the conclusion arrived at is that the oil lost in the treating reaction is a straight line function of the heat of the reaction. For 93% acid the amount of acid lost or consumed by reaction with the Solar oil varies 1 to 3·5% for treatments ranging from 1 to 7% by volume of treating acid. The amount of acid consumed is not proportional to the colour removed. The loss due to oxidation as indicated by the formation of SO_2 varies from 0·06 to 0·22% for treatments with 0·25–5·0% of acid by volume.

The question of nature of the reaction between the acid and the oil is discussed, and it is concluded that the physical or solution theory of acid treating is well supported by the experimental data, and that chemical reaction plays a very minor rôle Experiments on the reconstitution of the Solar oil, whereby the acid sludge is redissolved in the oil, show that the oil is not appreciably changed as a consequence of the

action of the acid. The reconstitution of the oil is accomplished by adding sufficient alcohol and benzol to the treated oil to dissolve all the sludge followed by washing to remove acid and distillation to remove the solvents. R. W. L. C.

199. Making of Low Pour Point Oils. E. R. Lederer and E. W. Zublin. *Oil and Gas J.*, 1930, **29** (32), 64, 101.

The three main processes in general use for removing wax from oil are cold pressing, centrifuging and filtering with a filter aid.

The use of selective solvents is not considered commercially possible at present owing to the high cost of solvents, such as butanol, some of which is inevitably lost in the process, but the fluid properties at low temperatures of oils prepared by this method are superior to those of any commercial product.

Many references to papers on wax formation are given, and it is thought that there are crystallization inhibitors, which when adsorbed on the surface of wax particles increase their surface tension and cause them to be surrounded by a protective film of oil. Thus the addition of a small percentage of inhibitor solvent, such as acetone, has a similar effect on crystallization as cracking, which is thought to destroy the inhibitors of tar and resin-like nature. The lowering of the temperature to a point where the inhibitor begins to change from true solution to colloidal state, results in a smaller wax particle size, because the inhibitor is more readily adsorbed in its colloidal state. This is said to be the explanation of the increased difficulty of dewaxing by cold settling when the temperature is lowered beyond a certain point. The boiling range, nature and concentration of inhibitors present vary in the case of each individual crude oil and it is most important to establish the best plant conditions for fractionating the distillates in order to secure the most efficient separation of wax. Accurate plant control should then be instituted. A description of the cold settling and centrifuge methods of dewaxing are given, together with views on the essential factors for most efficient operation. Inhibitors to prevent crystallization of wax must be added to neutral oils since crystalline wax cannot be satisfactorily separated by centrifuge.

In the Weir process a filter aid, Hyflo-Super-Cel, is mixed with the diluted and chilled oil at the rate of 1lb./gal. The mass is slowly agitated to prevent settling of solids and is forced through a filter. Data on residuum from Ranger crude is given in support of the view that if dilution is below a certain minimum the rate of filtration drops suddenly. This is thought to be due to clogging of the filter by small wax particles caused by the concentration of inhibitor and wax. Ease of filtration varies for different crude and the process is used in the laboratory as a method of examining crude oils.

Copious references to literature and patents on the subject of dewaxing are quoted. R. A. E.

200. Contact Filtration in Petroleum Refining. L. P. White. *Petr. Eng.*, 1930, **2** (3), 172, 174, 175, 178.

For the contact filtration of lubricating oil stock two main types of clay are available, the natural and the acid treated. General experience shows that 1 lb. of acid treated clay will do the same amount of work as 2–2½ lb. of natural clay. Against this it must be remembered that

the former is nearly 4 times as expensive as the latter. Natural clay usually contains about 10% of water—this, of course, varies considerably with the origin and handling of the clay. It, therefore, has to be dried to prevent foaming during contacting, but is not made perfectly dry, as it has been found that a small percentage of moisture, causing a slight blanket of steam over the reacting surfaces, reduces the tendency to oxidation. Two per cent. of moisture is generally left in the clay for this reason. Acid-treated clays generally contain 15% moisture which is generally not dried out, as this causes it to lose some of its efficacy. It must, therefore, be used carefully to avoid foaming.

Another difference between the natural and acid-treated clays is that the former gives a more porous filter cake, resulting in higher rates of flow, easier washing, etc. On the other hand, a longer filtration cycle may be used with acid clays owing to the smaller amount of clay necessary to do the work.

Figures are given for the relative rates of flow possible with acid and natural clay and for the relative costs of clay required for contacting Pennsylvanian Bright Stock. It is shown that in this particular case natural clay costs $140 per day, against $145–$170 for treated clay. The advantage of the former is balanced by the lower filtration capacity required, and consequently lower oil losses in discharged cake, and lower naphtha losses in the case of the latter.

Reactivation of clay is carried out when it is not economic to waste it. Burning is the most usual method, 10%–20% being usually lost as dust, etc. More careful handling is required for reactivating acid-clay than for natural clays.

A process for reactivating spent clay by solvent extraction is being developed and will be described in a later article. C. L. G.

201. What Meaning has the Edeleanu Refining Process for the User of Transformer, Switch and Turbine Oils? I., F. Foerster; II., F. Frank; III., F. Foerster and F. Frank. *Erdöl und Teer*, 1930, 6, 558–561, 574–576, 591–593.

I. This is a discussion and criticism of the views of F. Frank as expressed in a recent work, "The Edeleanu Refining Process in Relation to Transformer, Switch and Turbine Oils" (*cf. Erdöl und Teer*, 1930, Parts 21, 22 and 23; *Elektricitats wirtschaft*, 1930, *Part* 512).

Foerster disputes Frank's view that oils of American origin refined by the Edeleanu process are equal to or superior in quality to Russian oils refined by the usual acid process. Edeleanu processed oils are not suitable for electrical use, the process merely being of value for obtaining useful products from certain crudes of inferior value.

The tar-formation number and deterioration (in the presence of metals) methods are discussed and the requirements for transformer and turbine oils as regards tar-formation, acid-sludge, etc., values in different European countries are given.

II. A reply to Foerster under the same title. Further data with regard to test-oils quoted by Foerster are given.

Frank considers that an alternative source to the Russian of transformer oils is necessary to avoid monopoly.

III. The conclusion of the discussion; including reply by Frank.

E. B. F.

202. Stabilising Effect of Liquid Hydrocarbons on Electrical Contacts. L. Bruninghaus. *Ann. des Comb. Liquides*, 1930, 5, 635–650.

It has been shown previously by the author (*Ann. des Com. Liquides*, 1929, 4, 515) that very thin layers of oil, under the application of differences of potential of some tens of volts, acquire a conductivity analogous to that of metals. The experiments have now been repeated and extended with greater precision.

The apparatus used consisted essentially of a dividing machine which measured down to one micron or less, fitted with two electrodes, one fixed to the platform and one to the carriage of the apparatus. Difficulties were first encountered due to backlash of the carriage, etc., but these were largely eliminated by arrangement of helical springs and an accuracy of measurement of the order of 0·1μ thus obtained.

The potential difference employed was 110 v. and measurements were made either with a galvanometer with sensitivity of 10^{-9} amps., or with a milliammeter, a shunt of 20,000 ohms and a circuit breaker being arranged in the circuit. The electrodes employed were, in the first experiments, two brass spheres of 2 cm. diameter, but later cylindrical electrodes of hardened steel were used. The fixed electrode was 5 cm. long and 2 cm. diameter, and the moving electrode 3 mm. diameter. The contacting surfaces were slightly convex and were highly polished and carefully cleaned.

The first experiments were carried out with air between the electrodes. Between perfect contact and non-contact there is a region of unstable conduction of the order of 1 micron, when the galvanometer swings violently. There is no sign of sparking or arcing when the electrodes are examined with the microscope. The region of conductivity increases to 14 or 15μ when a drop of oil is placed between the electrodes.

Several series of experiments are described using both air and oil between the electrodes, leading to the following main conclusions:

1. On separating the electrodes from contact in oil, the oil retains a state of high conductivity till the distance between the electrodes is about 14 to 15μ, when the current ceases. On approaching the electrodes again the oil remains an insulator until contact has been established. The cycle can be repeated indefinitely.

2. With air the effect is much smaller, the conducting zone being only about 0·3μ. Irregularities occur with air for no apparent reason, however. This is not the case with oil between the electrodes. These irregularities appear to be quite real, but difficult to explain.

3. There is apparently a critical value "h" of the field for which conductivity in the medium is established and a strength "h^1" for which conductivity vanishes.

Using the same potential difference (110 v.) and a supplementary resistance of 100,000 ohms, similar results were obtained. Using the galvanometer it was found that, with oil between the electrodes, on progressively reducing the gap from 20μ, no indication was obtained till 4 or 5μ had been reached. From 4 or 5μ to 0 the indications were unstable. At 0μ contact was established. On separating again the highly conductive state is maintained till 15μ has been reached.

It is thus concluded that hydrocarbon oils have a marked stabilising effect on the making and breaking of contacts and that these phenomena are of great importance in the construction of sensitive and accurate relays. E. B. E.

203. Some Aspects of Grease Lubrication. Effect of Composition and Properties upon Lubrication. H. R. Mathas. *Petr. Eng.*, 1930, 2 (3), 85, 86, 88.

The choice of the most suitable type of lubricant for a particular purpose requires consideration of the pressure, speed and temperature of the moving parts involved, the method of lubrication, design of parts, kind of rubbing surface and special operating conditions. Wherever possible oil should be used, but for certain purposes greases are superior in reducing friction and minimising wear. A true grease is usually defined as a solid or semi-solid lubricant in which a soap is incorporated in petroleum lubricating oil. The two chief types of soaps are sodium and calcium (or lime) soaps; the former yielding a high melting, fibrous grease, not resistant to water, and the latter a lower melting smooth grease, insoluble in water. Magnesium, zinc, aluminium and lead soaps also find practical application in grease making. Fillers are often introduced, solely to reduce the cost of the grease, *e.g.*, graphite, mica, clay, soapstone, wood fibre, asbestos, ground cork, etc. The first three named have a certain lubrication value in specific instances, but should not be used in anti-friction bearings, while the remainder of the fillers have no lubricating value whatever. Greases are usually graded by their consistency. This is often determined by the "feel" of the grease but can be estimated scientifically by the consistency test drawn up by the A.S.T.M. This is determined on the "worked" or "unworked" grease, preferably the former, since the consistency varies with the physical treatment or handling the grease has received. "Working," or forcing the grease through a perforated plate or hose of a pressure gun brings it to maximum softness.

Some greases are manufactured to a final alkaline reaction, others to acid, but in the case of the latter only fatty acids, which have no corrosive action on metals, are present. Perfume is often added to mask the disagreeable odour of rancid or insufficiently refined oils used in the making of the grease, so that such greases should be regarded with suspicion. The viscosity of the oil used in the grease is also of great importance.

A list is given of the properties and applications of the different types of greases. For high temperature work, such as mill or railroad grease, fibre or sponge greases, sodium fats or fatty acids or mixtures are used, while for cup greases calcium fats or fatty acids are more suitable. For axle and gear grease calcium and rosin soaps are used, and for gear shields a heavy bituminous oil. C. L. G.

204. Crane Lubrication. E. H. Thompson. *Mech. Eng.*, 1930, 52, 1059–1062.

The high cost of maintenance and delays due to lubrication are mainly caused by an insufficient supply of oil and grease.

It has been found advantageous to adapt for the crane equipment an oil of medium viscosity and of naphthene type, *viz.*, 500 sec. at

100° F. (Saybolt). This oil has proved suitable for all seasons and conditions of use.

The loss of hoist armatures on obsolete types of monorail cranes due to oil leakage was overcome by placing an oil-thrower between the oil cellar and the commutator.

The tendency of the dry wooden blocks to seize the clutch on obsolete cranes fitted with a cone type brake on the hoist motor shaft was overcome by fitting grease caps in the stationary clutch. W. A. W.

205. Enlargement of Grozni Paraffin Wax Plant. A. Dembo. *Azerbaijan Oil Ind.*, 1930, No. 10, 21–30.

The installation and working of the process was described by A. Ablovaski in the *Oil Industry*, 1929, No. 4. The plant began to operate in 1927. The progress was as follows:—

	1927–1928. Tons.	1928–1929. Tons.	1929–1930. First 6 months.
Paraffin Mazut used	103,176	155,363	100,315
Paraffin Wax Output	4,137	6,014	4,302
Losses (% of Mazut)	5·29	2·4	2·07
Fuel used (% Mazut)	16·7	10·4	9·02

Self cost of the paraffin wax:—

	1927–1928. Rubs./ tons.	1928–1929. Rubs./ tons.	1929–1930. First 6 months.
Paraffin wax for matches ..	334·0	175·8	135·0
White Paraffin Wax	402·0	208·2	173·1

It is proposed to increase the scope of the operations as follows:—

To be used:—	1930–1931.	1931–1932.	1932–1933.
Paraffin crude	—	420,000	840,000
Mazut	331,500	210,000	210,000

Output.	1930–1931.	1931–1932.	1932–1933.
Solar distillate	22,650	115,170	180,336
Filtrate I.	136,044	—	—
Paraffin Scale 2	—	27,984	38,115
Slop Wax	25,029	46,200	97,800
Paraffin gudron	125,250	135,450	202,950
Heavy benzine	—	96,600	193,200
Kerosine distillate	—	84,000	168,000
Spindle Oil	—	31,720	42,768
Machine Oil	—	45,131	60,588
White Paraffin Wax	16,370	24,616	33,105

The products to have the following qualities—Paraffin wax (white, non-oily and not discoloured in sunlight) m.pt. (Shukoff), 52°–53° C. Spindle oil, sp. gr. at 15° C., 0·885; viscosity (Engler) at 50° C., 2·3°;

pour point (Holde), –8°C. Machine oil, sp. gr. at 15° C., 0·905; viscosity (Engler) at 50° C., 6·0°; pour point (Holde), –10° C.

Complete estimates of the costs of production and values of the plant year by year up to 1933 are given in the original papers. P. A.

206. New Device for Sweating Paraffin. Laboratory of Shell Group. *Petr. Zeit.*, 1930, **26**, 1217–1220.

The apparatus consists of cooling and sweating vessels arranged one above the other in a small space, either in couples or in sets of fours. The crude paraffin is brought to the desired temperature in a space where the low-melting constituents are sweated. The following are the most important advantages of the new device:—(*a*) different initial substances can be treated simultaneously in one and the same arrangement, (*b*) the process is continuous, (*c*) the vessels can be filled during the progressive sweating, and the sweating vessels can be cleaned without disturbing the operation, (*d*) the rotary velocity of the sweating vessels can easily be adjusted to suit the type of material treated and the sweating oil can be totally fractionated, (*e*) the apparatus is labour-saving and can be worked by one man. Several sectional diagrams of the apparatus are given. W. S. C.

207. Process and Apparatus for the Reclamation of Used Lubricating Oil. Aktiebolaget Separator, Sweden. E.P. 318,626, 4.9.29.

Used lubricating oil is liberated from light oil fractions, water and solid impurities, and given the desired viscosity by repeated centrifuging at a temperature of from 90° C. to 120° C., in a separator provided with arrangements for producing a strong current of air in the collecting vessels of the separator, any impurities remaining in the oil being then removed either by filtration or treatment with bleaching material and subsequent centrifuging, or filtration, or both. The light fractions distilled off during the centrifugal process are condensed and collected in an apparatus connected to the separator. H. E. T.

208. Apparatus for Cleansing Oils. Preston, Stallard and Co., Ltd. E.P. 337,981, 8.11.29.

An apparatus is described for cleansing waste or dirty lubricating oils and comprises a tank to contain the oil to be treated, an electrical heater associated with the tank, and means to circulate through the oil a liquid having a sp. gr. substantially different from that of the oil. In this type of apparatus the circulation of water or other cleaning fluid causes the fine particles of carbon and other suspended impurities in the oil, to coalesce and form aggregates, which on cooling the oil will readily separate by gravitation. A delay action switch is provided and may be set so that the heater and circulator remain in action for the proper length of time, after which they are automatically cut out. The delay switch is controlled by an adjustable clockwork mechanism. H. E. T.

See also Abstract No. 194.

Fuel Oil.

209. Industrial Uses of Fuel Oil. I. Lubbock. *J. Inst. Fuel*, 1930, 4, 90–112.

The applications of fuel to a large number of industrial processes, with particular reference to the glass, ceramic, metallurgical and cement industries are described.

Although dearer than coal, economies can be effected from the use of fuel oil which more than outweigh its direct cost. Figures are given in particular for the cost of an oil-fired galvanising bath as compared with a coke-fired bath, and it is shown that while the cost of coke is 30s./ton and fuel oil 65s./ton, the production can be doubled when fuel oil is used, and the cost of production less overheads brought down from 1s. 10d. to 1s. 5½d. per ton.

In connection with the conversion of glass furnaces from oil producer gas firing to oil firing, it is interesting to note that with oil fuel more B.Th.U. can be fed to the furnace per foot of combustion space, while higher flame temperatures can be reached. As a result the combustion space can be reduced, leading to economy in fuel consumption. The height of the regenerator can also be reduced owing to the injector action of the burner itself, while there is less silting up of the regenerator packing, due to the greater cleanliness of the fuel.

Oil is being increasingly used in metal smelting. The chief advantage in the use of fuel oil for tin is the reduction of metallic losses in the waste gases. It was also found possible to use oil firing for melting pure nickel (pouring point 1600° C.) in a particular case where an electric furnace failed to do the work. The use of oil fuel in sintering, forging furnaces, tube welding, wire annealing, galvanising, salt baths, heat treatment furnaces, core drying ovens, roll heating and bright annealing, has also resulted in considerable economies.

Many potteries and brick works are being converted to oil firing, resulting in better ware, shorter firing periods and saving in saggar wastage. It is interesting to note that the Sèvres Pottery Works has used oil for many years.

Road mixing plants are in many cases now oil fired, the chief advantage lying in the reduced consumption of fuel, since it is not necessary to keep the fires going when the plant is not in use.

Oil fuel is used extensively in America for central heating, and its use in England is increasing. The main advantages are elimination of smoke, cleanliness of boiler houses, ease of delivery, automatic regulation of temperature and in many cases direct fuel cost reduction.

Bakeries and cookeries are now being equipped for oil firing. Particularly in smaller bakeries conditions are rendered very much more comfortable for the workers, while the disadvantage of the presence of solid fuel, ash, clinker and cinders in proximity to the dough is removed.

Details are given of the applications of fuel to many other industrial processes with a large number of scale drawings of furnaces and photographs of oil fired plant. C. L. G.

210. Oil Fuel Burners and Supply Apparatus Therefor. Shell Mex, Ltd. I. Lubbock and J. R. Joyce. E.P. 339,314, 5.9.29.

The invention relates to automatic burners, *i.e.*, those in which ignition is intermittent.

A draught tube houses the burner, controlled by a needle valve in a jet, and carries near the burner end a reservoir containing an electrical immersion heater and a controlling immersion thermostat, by which the fuel is kept warmed, and circulates round the nozzle by thermo-siphonic action when the burner is not operating. This reservoir is supplied with fuel from the main tank by a pump driven from the same shaft as the air blower and has a relief valve to prevent any excessive rise of pressure, and a leak device at the bottom which allows dirt and water to be ejected and permits "breathing" due to the expansion of the oil. Ignition is effected by a high tension spark between two electrodes, which are cleaned each time the burner starts or stops, by a wiper operated by a link mechanism from a piston. The latter is attached to the spindle which carries the needle valve controlling the burner jet, and is operated by the fuel pressure. A vane to control the air supply is also operated by this piston, the air supply being cut off when the burner shuts down. J. M. F.

Fluxes and Asphalts.

211. Road Laboratories in the U.S.A. P. E. Spielmann. *Roads and Road Constrn.*, 1931, **9**, 9–10.

American State Road laboratories are primarily occupied with routine testing of all road construction materials, iron for bridges, paints, etc., this being required where Federal Aid is granted. The Bureau of Public Roads has its stone and concrete laboratory in Washington, where mineral aggregate is examined and where certain field sorting tests for strength have been elaborated. The Arlington Experimental Farm (nr. Washington) has carried out analyses of subgrade soil and interpreted the results in terms of road making practice, the work having come to fruition after ten years. As a result, recommendations can be made on the type of pavement most suitable for construction over the type of subgrade identified in the laboratory. The effect of constituents of concrete on its strength is being examined. Experimental wear of surfacings on a circular track have shown the destructive effect of unsprung weight and the protection afforded to road and vehicle by pneumatic tyres.

The University of Michigan, *inter alia*, examines asphalt mixtures by the Hubbard-Field machine and also studies the effect of low temperatures on subsoils and road surfacings. The Pennsylvania Laboratory is the latest, and all road constructional material (including sewerage pipes, road signs, etc.) are examined. It has chemical, physical, sand and cement laboratories. Noisy machinery is housed in sound-proof rooms; weathering tests are carried out on the roof; it possesses compression machines working up to 400,000 lb.; and a constant temperature water supply is available in many positions.

C. I. K.

212. Highways of the U.S.A. Part III. Road Mixed Bituminous Construction. T. W. Allen. *Roads and Road Constrn.*, 1931, **9**, 12-14.

In view of the relatively small funds available for road work, it was found necessary to devise roads not costly to build and maintain, and suitable for the vast mileage of intermediate roads carrying 300 to 1000 vehicles a day. A promising development carried out on a vast scale was the road-mixed bituminous construction, which consists of three steps. (1) The securing of a 3 in. course of loose material (sand or gravelly soil, gravel or broken stone) by scarifying the original surface and/or adding new material. (2) Sprinkling or spraying this loose material with a fluid bitumen (asphaltic oil) sufficient to coat all particles. This is applied hot by a pressure distributor (1,000 gallons capacity) in about three applications, each being followed by passage of a spring tooth harrow, and/or a double disc harrow. (The finer the material the more binder required.) Mixing is continued by blade graders until the binder is distributed uniformly through the mass of aggregate. The surfacing is placed in a windrow and spread in a true cross section of desired depth. (3) Consolidation is usually done by traffic, blade graders being used in the meantime to maintain a smooth surface. Heavy rains followed by cloudy weather necessitate additional mixing and sometimes reblading into windrows, drying out and relaying.

Modifications of the above are many. One is making repairs to the old surface; removal of dust; application of a priming coat; spreading of stone or gravel; application of binder followed by blade mixing and final spreading on the road. Small stone is then broomed into the roads, followed by more binder and finally stone chips with rolling.

Costs range from $2,000 to $10,000 a mile according to conditions. This can be reduced by accumulated experience. C. I. K.

213. Treatment of Bitumen, etc., for Electrical and Other Uses. D. Anderson and Son and R. O. Child. E.P. 339,470, 25.11.29.

Bituminous material is subjected in a normal or heated condition to the action of a suitable solvent and the resultant solution added to a quantity of colloidal silicic acid. The solution may be decanted or subjected to distillation for the purpose of imparting thereto the desired degree of viscosity. W. H. T.

214. Tar for Use on Roads. Thermal, Industrial and Chemical Research Co. and C. O. Condrup. E.P. 339,640, 6.8.29.

A satisfactory road tar of strongly adhesive nature is obtained by adding a small proportion of H_2SO_4 to a crude tar. The mixture is agitated at a suitable temperature (120°–189° F.), neutralised and dehydrated, finally volatilising the light oils if necessary to obtain the desired consistency. W. H. T.

See also Abstract No. 298.

Special Products.

215. Electrostatic Recovery of Acid from Sludge. *Petr. Times*, 1930, 24, 1000.

Hot air is passed through acid sludge to vaporise acid and resultant gases, after partial cooling, are passed through Cottrell electrical precipitators at a suitable rate. 95% of the suspended acid mist is recovered at a concentration of 41–62%. Power consumption for 50 tons 66° Bé. acid per day is 12–15 k.w. The Cottrell electrical precipitator is described in some detail. H. G.

216. Active Carbon from Pitch. *Chem. Trade J.*, 1931, 88, 26.

Since there is a surplus of pitch in Manchuria, the Central Laboratory of the South Manchurian Railway Co. have endeavoured to convert pitch from the Anshan Ironworks into a wood preservative by extraction with organic solvents under pressure in the presence of catalysts. On the laboratory scale they have obtained products which can be used for this purpose when mixed with creosote. If the residue from solvent naphtha extraction ("free carbon") is mixed with an equal weight of anhydrous potassium sulphate, and the mixture calcined in an electric furnace, a highly porous pulverulent decolorising carbon is obtained. Comparative tests showed the efficiency of this carbon to be greater than that of animal charcoal and a number of proprietary decolorising carbons. R. A. E.

217. Methyl Alcohol as an Anti-Freeze. *Chem. Trade J.*, 1930, 87, 631.

The U.S. Bureau of Mines has investigated the use of methyl alcohol for anti-freeze purposes and a preliminary report has just been issued. The findings and recommendations are—(1) There is no danger of poisoning from the reasonable use of methanol as an anti-freeze; (2) evidence of methanol poisoning through absorption through the skin is rare and inconclusive; (3) occasional exposure to methanol is not dangerous, although continued exposure to high concentrations is; (4) all methanol is poisonous when taken internally, regardless of its purity or method of manufacture; (5) anti-freeze methanol should be coloured distinctively; (6) all methanol sold for anti-freeze use should be labelled to call attention to the risks of its misuse. Further studies are being made. R. A. E.

218. Metallic Ships' Bottoms and Means of Preserving them from Rust. H. Masseille. *Peintures, Pigments, Vernis.*, 1930, 7, 1193, 1219, 1244. *Oil and Colour Trades J.*, 1930, 78, 512.

The author has sought the comparative values of turpentine and white spirit as paint thinners. On account of its cheapness, and in some cases its superiority, white spirit has been preferred to turpentine by some French Government administrations.

Paints were made according to the following formulæ, one set containing turpentine and the other white spirit as the thinner:

	Ordinary.	Semiflat.	Flat.
Linseed Oil %	32	20	10
Siccative %	2	2	2
Zinc White %	60	68	60
Thinner (Volatile) % ..	6	10	28

The tests were carried out by applying each paint to (1), a board of pitch pine (kept in an interior); (2), a plate of sheet iron left exposed to the weather; (3), bands of surface in the cabin of a cruiser painted side by side.

No differences were observed on account of ease of application or covering power, but after a few months the white spirit paints had turned slightly yellow, whereas the turpentine paints had not. Only in the case of the "ordinary" grades of paint were differences noted in rapidity of drying—the turpentine paints drying more quickly, due to the energetic oxidation possible. The white spirit paints apparently dried by evaporation of the thinner.

Storage of the paints in hermetically sealed tins for six months showed that the white spirit paints had become noticeably thicker and the colour visibly affected more than in the case of the turpentine paints. Masseille also reported that a paint made with kerosine as the thinner had become much thicker and the colour had turned creamy.

Masseille does not drawn any definite conclusions from the results, though the implications are obvious. C. I. K.

219. French Tests With Turpentine and White Spirit. L. A. Jordan. *Oil and Colour Trades J.*, 1930, 78, 1848.

In reply to H. Masseille (see preceding abstract), it is stated that great efforts have been made in France and U.S.A. to establish the value of turpentine in paint and varnish. Actually the value of turpentine is not to be questioned. Turpentine producers in France are vigorously agitating against the use of white spirit and demand prohibition of its importation. Apart from the relative values of both thinners, the question has become chiefly economic, with the result that the use of white spirit has increased enormously, and with justification.

Whilst no specifications of the oil drier were offered, it seems that Masseille used a turpentine intermediate between types 1 and 2 of B.E.S.A. specification. The white spirit had the following properties—sp. gr. at 15° C., 0·77; I.B.P., 88° C.; distillation, at 120°, 15%, at 130°, 39%, at 140°, 61%, at 150°, 75%, at 160°, 85%, at 170°, 91%.

Jordan contends that it could scarcely be called a white spirit in an English commercial sense (cf. B.E.S.A. specn. type 1. White spirit).

It was to be expected that turpentine paints should give good results, but not that the white spirit paints should be so invariably bad.

Circumstances can and do arise in manufacturing technique when, as an alternative to turpentine, white spirit has definite and positive advantages, not the least of which is consistency in quality and composition.

Isolated and premature results from experiments which are technically weak should not be used as a basis of general conclusions.

C. I. K.

220. Catalytic Oxidation of Carbon Monoxide. J. C. W. Frazer. *J. Physical Chem.*, 1931, **35**, 405.

A short summary is given of recent work carried out on the catalytic oxidation of carbon monoxide by means of metallic oxide catalysts. The best catalysts for promoting the oxidation are, manganese dioxide and silver oxide, manganese dioxide and copper oxide, and manganese dioxide alone. It is evident that the manganese dioxide is the more active of the two constituents. Seventeen oxides were prepared and examined electrolytically, but none of them had the same high activity as the above three, although ferric oxide showed some activity below 100° C. H. E. T.

221. Influence of Various Catalysts in Promoting the Oxidation of Methane by Means of Copper Oxide. J. R. Campbell and T. Gray. *J.S.C.I.*, 1930, **49**, 447T–450T.

The apparatus used consisted of an aspirator bottle containing the gas sample, the gas being delivered at constant rate through a sulphuric acid bubbler to the combustion tube containing known weight of copper oxide and fitted with pyrometer accurate to ±5°.

The catalysts used were deposited on the copper oxide, which had been activated by alternate reduction and oxidation, as follows—$CuO+CuCl_2$ mixture by mixing the copper oxide with a suspension of copper chloride in water and drying; $CuO+V_2O_5$ by ignition of copper oxide soaked in ammonium vanadate; $CuO+CoO$ and $CuO+NiO$ by igniting copper oxide impregnated with the corresponding nitrates; $CuO+MnO_2$ by igniting copper oxide suspended in freshly precipitated manganese dioxide.

Cuprous chloride is by far the most efficient catalyst but its efficiency falls off if it is heated above its melting point. At temperatures above 650° C. free hydrogen reduces the chloride, so that in carbon and hydrogen estimations care must be taken not to exceed this temperature unless adequate precautions to ensure the oxidation of this hydrogen chloride are taken. Cuprous chloride, however, may be regarded as the ideal catalyst for Dumas' method for nitrogen determination.

The most efficient of the other catalysts is cobalt oxide, the remainder in order of efficiency being manganese dioxide, nickel oxide and vanadium pentoxide. The effectiveness of the catalyst in promoting the oxidation of methane by means of copper oxide is a function of the amount of catalyst present in the case of vanadium pentoxide, and this probably holds for the others. A. R. S.

222. Freezing and Flow Points for Glycerol, Prestone, Denatured Alcohol and Methanol. J. C. Olsen, A. S. Brunjes and J. W. Olsen. *Ind. Eng. Chem.*, 1930, **22**, 1315, 1316.

The freezing and flow points of liquids used as anti-freeze materials have been redetermined. It is suggested that the flow point gives the minimum temperature at which a liquid may be used for cooling radiators. In some cases the flow point is considerably lower than the freezing point; in other cases there is not much difference. The freezing points and flow points of prestone, methanol, denatured alcohol and glycerol, at various dilutions, are given in a table.

The glycerol sample, sp. gr. at 20°/20° C., 1·2546, satisfied the tests for purity as compared with U.S.P. glycerol Prestone, a commercial grade of ethylene glycol, was coloured deep green and had sp. gr. at 20°/20° C., 1·1095. Thermal alcohol, completely denatured, formula No. 5, 188 proof, had sp. gr. at 20°/20° C., 0·8197. A synthetic methanol sample was used. W. D. S.

223. Preparation, Properties, and Uses of Glycerol Derivatives. Part II. Acetals, Amines and Resins. A. Fairbourne, G. P. Gilson, D. W. Stephens. *J.S.C.I.*, 1930., **49**, 1069–1070.

Acetals, which are cyclic ethers of glycerol, are produced in theoretical yield by the condensation of glycerol with aldehydes or ketones in the presence of a small amount of hydrochloric acid as catalyst. The preparation and physical properties of methylidene-, ethylidene-, isopropylidene-, benzylidene-glycerol are given. These cyclic compounds have excellent solvent properties but do not possess the special qualities of stability associated with the normal ethers of glycerol. The lower members are miscible in all proportions with water, the solubility decreasing with increase of molecular weight.

Amines derived from glycerol may be readily obtained by the action of ammonia on the chlorohydrins. The properties of these bases may be roughly described as those of a non-volatile ammonia. They combine readily when warmed with fatty acids to produce soaps soluble in organic liquids, and therefore capable of use in dry-cleaning preparations.

An important advance follows in the manufacture of resins from glycerol ethers as excess of the latter, unlike glycerol, is not harmful to the lacquers. No commercial application appears to have been developed for the great variety of resins obtainable from the glyceryl-amines. Their preparation and properties are described.

W. D. S.

224. Covering Carriers with Bituminous Substances. H. D. Elkington, Assr. to N. V. de Bataafsche Petr. Mij. E.P. 312,909, 17.5.29.

A process is described for covering carriers with bituminous substances for preparing moisture-proof products (asphalt paper). The carrier is covered with bituminous substance in the form of an emulsion, the bitumen being reduced in viscosity during any of the stages of manufacture below 756 sec. (Red. II.). This reduction is effected either by heating the bituminous paper above 120° C. or by selecting a bitumen which has a low viscosity at 80°–120° C. but is not sticky at normal temperature. Viscosity can also be reduced by the addition of materials such as paraffin wax, resin or naphthalene. The paper fibre mass is glued with resin-glue and alum before the addition of asphalt emulsion, the viscosity of the latter being reduced in the presence of a non-oxidising medium. W. H. T.

225. Catalytic Processes for the Hydrogenation and Dehydrogenation of Organic Liquids. E. J. Du Pont de Nemours and Co. E.P. 313,093, 6.6.29.

The invention relates particularly to syntheses of organic compounds by the catalytic hydrogenation under pressure of oxides of carbon (*e.g.*, carbon monoxide). The catalyst comprises difficultly reducible basic

and acidic oxides together with form 0·25% to 5·0% of an alkali metal compound calculated as the element, the acidic oxide being in excess of that necessary to form a neutral compound with the basic oxide (*e.g.*, zinc oxide and chromium oxide—ratio less than 2:1). The catalyst may contain about 5% of its weight of anhydrous sodium chromate. The reaction is effected at a temperature of about 475°–500° C. and under a pressure of from 100–1000 atm. (*e.g.*, 275 atm.). W. H. T.

226. Catalytic Manufacture of Synthetic Acetic Acid. Soc. Française de Catalyse Généralisée. E.P. 338,329, 26.1.29.

Synthetic acetic acid is manufactured catalytically by hydrogenation of carbon monoxide in the presence of catalysers containing nickel, cobalt, chromium, iron or copper. As catalysers the various forms of phosphates, silicates and borates, of these metals are used either alone or mixed together with any other metal, oxides, carbonates and, generally speaking, with any other salt. The reaction is carried out at a temperature between 500° and 550° C. and at a pressure between 450 and 600 atm. W. D. S.

227. Manufacture of Anhydrides of Organic Acids. H. D. Elkington and N. V. de Bataafsche Petr. Mij. E.P. 338,507, 9.8.29.

Aliphatic acid anhydrides are prepared from monobasic aliphatic acids and monoketones by allowing mixtures of these compounds to react at elevated temperatures (above 600° C.) and at ordinary, increased or reduced pressures. The mixture is passed through tubes containing filling or contact masses comprising copper, yellow brass, or aluminium sulphate, and under the chosen conditions of temperature and pressure. Instead of monoketones, substances which give rise to monoketones under the conditions of the reactions may be employed. Several examples are given, such as acetic anhydride from acetic acid and acetone, and from acetic acid and isopropyl alcohol. Large amounts of methane and carbon dioxide occur in the gaseous products. W. D. S.

228. Manufacture of Ketones. H. Dreyfus. E.P. 338,518–9, 19.8.29.

A method for the manufacture of aliphatic ketones (especially acetone) is described. The vapours of an aliphatic alcohol containing at least two carbon atoms are mixed with oxygen and submitted to the action of high temperatures in the presence of one or more oxides, hydroxides, carbonates, or other compounds (particularly weak acid salts), of the alkali earth metals. The temperature should be between the limits 250–700° C., and especially between 350° and 550° C. The process may be performed in the presence of relatively large amounts of aliphatic acids if desired. An example is given in which a mixture of ethyl alcohol vapour, oxygen and acetic acid vapour in about the proportions 1:5:1 by volume, is passed through a copper tube filled with a catalyst composed of calcium oxide, calcium silicate or augite $CaMg(SiO_3)_2$ and maintained at a temperature of about 430–480° C. There results a copious yield of acetone.

In place of oxygen, steam may be used in admixture with the alcohol and aliphatic acid vapour. An example gives a mixture of ethyl alcohol vapour, steam, and acetic acid vapour, in about the proportions 1:3:3 by volume. W. D. S.

229. Polymerisation of Diolefines. J. Y. Johnson, Assr. to I. G. Farbenind, A.-G. E.P. 338,534, 17.8.29.

A process is described for the production of polymerisation products of diolefines. The polymerisation is carried out in the presence of alkali or alkaline earth metals in the form of particles of a uniform size. The average diameters of the various particles should not differ by more than 10%. W. H. T.

230. Manufacture of Alkyl Halogen Compounds. N. V. de Bataafsche Petr. Mij. E.P. 338,742, 27.11.30.

It is difficult to manufacture chlorinated paraffins by exposing a mixture of chlorine and the paraffin to sunlight, and at high temperatures explosions are likely to occur. These difficulties have been overcome by mixing the chlorine or bromine and the hydrocarbon in the dark and at a low temperature but at which both components are in vapour form. Thus a premature reaction between the components is avoided and the mixture can be heated to an elevated temperature without the use of sunlight, daylight or artificial light. There is a critical temperature, varying with each paraffin, at which the monohalogenated paraffins may be easily obtained. The temperature for instance lies between 380° C. and 400° C. for methane, between 360° C. and 380° C. for propane, between 330° C. and 380° C. for butane, and between 250° C. and 330° C. for pentane. Moreover it has been found that a large excess of hydrocarbon over halogen is not necessary. W. D. S.

231. Manufacture of 1:3-Butylene-Glycol by Hydrogenising Paraldol. I. G. Farbenind. A.-G. E.P. 338,807, 30.1.30.

Paraldol is converted into 1:3-butylene-glycol by treating it with hydrogen in the presence of a hydrogenating catalyst. The process is carried out by dissolving the paraldol in water and adding metallic nickel in a finely divided form on pumice. The whole is hydrogenised at a temperature between 30° C. and 50° C. and a pressure of hydrogen between 20 and 25 atm. in a revolving drum provided with a bucket or scoop stirrer for the purpose of intimate mixing. After filtration, the 1:3-butylene-glycol, b.p. 206–207° C., is fractionated out and obtained in a nearly quantitative yield. W. D. S.

232. Production of Gas from Heavy Hydrocarbon Oils. A. S. Ramage. E.P. 338,904, 24.8.29.

Combustible gas and aromatic hydrocarbons are produced from the vapours of normally liquid paraffin hydrocarbons which are contacted first with ferric oxide and then with finely divided iron at progressively increasing elevated temperatures and in the presence of progressively increasing quantities of steam. The ferric oxide is maintained at a temperature of about 900°–1000° F. and the divided iron at a temperature from 1150°–1500° F. The vapours are contacted with the ferric oxide in the presence of 1-2% of steam and the resulting gases admixed with 10–20% of steam before being passed over the divided iron at 1150° F The vapours produced are mixed with 20–40% of steam and passed over the iron at 1300° F. W. H. T.

233. Making Gases from Hydrocarbon Materials. A. S. Ramage. E.P. 338,905, 24.8.29.

This invention relates to the production of permanent gases from hydrocarbons by passing the latter together with steam into contact with heated iron. The hydrocarbons are olefine compounds substantially free from paraffin hydrocarbons in gaseous or vapour form. The gases produced consist principally of carbon monoxide, hydrogen and methane. W. H. T.

234. Polymerisation Products of Diolefines. J. Y. Johnson and A. Carpmael. Assrs. to I. G. Farberind. A.-G. E.P. 339,135, 20.12.29.

The invention relates to improvements in the manufacture of polymerisation products which may be highly viscous, plastic and/or elastic, by the polymerisation of materials such as butadiene, isoprene or dimethyl butadiene, etc., in the presence of an alkali metal. The reaction is carried out in the presence of a low boiling aliphatic ether in an amount exceeding 25% by weight of the diolefine employed. W. H. T.

235. Manufacture of Polymerisation Products of Diolefines. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 339,243, 30.8.29.

Valuable products of diolefines may be obtained by polymerising the diolefines with the aid of non-ionogeneously bound metals or alkaline metal earths. By a non-ionogeneously bound metal is meant metal in the free state, as alloy with another metal or in the state of its compounds with organic radicles. The latter should not be capable of ionization, and as examples sodium ethyl, potassium diphenyl methylmethyl may be quoted. By adding the polymerising agent gradually or in at least three portions during the course of the polymerisation, excellent products are obtained. Solvents such as other hydrocarbons or ethers may be present and temperatures ranging between 0° and 100° C. are employed. Examples of the process are given for isoprene, butadiene and dimethyl butadiene. W. D. S.

236. Manufacture of Acetone. H. D. Elkington and N. V. de Bataafsche Petr. Mij. E.P. 339,491, 6.12.29.

The conversion of isopropyl alcohol into acetone is carried out by passing the alcohol in vapour form, with or without admixture of other gases or vapours, through a liquid medium, which is heated to a temperature between about 300° C. and 700° C. As liquid medium, molten lead is preferably used, but other molten metals, alloys, or fused salts may be used. The medium selected should not appreciably attack the isopropyl alcohol or acetone, nor should it give rise to undesirable side-reactions. Partially concentrated solutions of isopropyl alcohol may be used directly without previously freeing the same from water. By suitable regulation of the gas velocity, an almost complete conversion of the alcohol into acetone may be attained. W. D. S.

237. Process of Reclaiming Residues. A. E. Becker and R. G. Sloane, Assrs. to Standard Oil Development Co. U.S.P. 1,785,242, 16.12.30. Appl. 28.11.23.

A process is described in which residues containing organic acid compounds are worked to yield useful products.

The residues, treated with caustic soda, are thinned with a relatively low-boiling thinning agent, *e.g.*, naphtha, and water-washed. The naphthenates are recovered from the wash water and the residue treated with 66° Bé H_2SO_4 at the rate of about 50 lb./brl. The sludge is removed after settling, treated with caustic soda solution, washed, and the naphtha is removed by distillation. W. S. C.

See also Abstract No. 188.

Refining and Refinery Plant.

238. New System for Saving Tank Vapours. L. G. E. Bignell. *Oil and Gas J.*, 1930, **29** (32), 62, 63.

Many systems for preventing loss of vapours due to "breathing" of tanks have been used in the past. Pressure relief valve systems are limited by the pressure which storage tanks are capable of withstanding. The method of collecting expelled vapours in a cloth balloon is attended by mechanical difficulties. J. H. Wiggins has developed a new method of storing expelled vapours in a steel balloon. This balloon can best be described as a shallow tank of wide diameter, the top and bottom acting as diaphragms which are free to move either up or down as vapour enters or is expelled. The structure is suitably guyed to withstand wind load and provision is made for drainage of the upper diaphragm. Snowload is carried until the snow melts, and if the pressure becomes higher than is desired a pressure operated valve releases the gas.

The balloon is connected to the vapour space of the storage tanks by large diameter piping, in hydraulic gradient to prevent plugging. There are no valves between the tanks and the balloon, which is itself vented by extra large pressure and vacuum valves operated by the diaphragms. This absence of valves on the tanks constitutes a factor of safety, since the balloon will rupture itself before enough pressure or vacuum is placed on the tanks to injure them.

The system has an advantage over gasoline recovery systems because of the elimination of labour, operating and pumping charges.

Results of tests are given showing the saving of gasoline effected. Approximately 87% of the vapours expelled were saved during one test of 2 months on a tank partly filled with gasoline and connected with a steel balloon.

It has also been shown that the system seldom takes in any air, suggesting the possibility of using a non-inflammable gas, such as CO_2, thus eliminating the fire hazard and minimizing the corrosion of the interiors of tanks and balloon, by the exclusion of oxygen. R. A. E.

239. Influence of Water Vapour on Rectification. K. Kostrin. *Azerbaijan Oil Ind.*, 1930, No. 10, 99–103.

The water vapour is harmful to rectification. The effect is not due to water vapour as such, however, but is caused by condensation of the vapour into water. When the temperature of the rectifying column is above 100° C. it is possible to use water spraying with satisfactory results. Naturally, the effect of water vapour on the velocity of vapours in rectifying columns must be taken into account when designing the apparatus. P. A.

240. Sinclair Enlarges Wellsville N.Y. Plant. A. E. Mockler, *Oil and Gas J.*, 1930, **29** (30), 96, 97, 192.

The Sinclair Refining Co. is on the verge of completing extensions and modernization of their Wellsville N.Y. plant, which will make it the world's largest and most modern refinery handling Pennsylvanian crude. The capacity of the plant is 10,000 brls. of crude per day, the main feature of the refinery being a new 7,500-brl. Foster Wheeler crude still with a 155 ft. tower yielding 7 cuts simultaneously. Operation is controlled from the receiving houses by means of de Florez remote control valves. Work is also being commenced on a cracking plant designed by the Sinclair engineers, with a de Florez heater, capacity 2,000 brls. per day. The refinery also contains rerun batch still, lub. oil contact filtration plant and dewaxing plant, and the usual boiler house and barrelling plant, storage, handling and pumping facilities.

The lub. oil stock from the Foster Wheeler still is contact filtered three times at a high temperature, mixed with naphtha, cooled to 60° F. below zero, and centrifuged in Sharples super-centrifuges running at 15,000 r.p.m. This low temperature is necessary to give the finished oil its unusually low pour test. The dewaxed oil is then run to an 8000-brl. Foster Wheeler rerun still from which lub. oil is taken as a finished product. Both the crude oil and lub. oil rerun stills are fired with powdered coal, although arrangements are available for oil firing. The boiler house is equipped with 3 stoker-fired boilers of 125 lb. pressure, and 2 automatic pulverized coal-fired boilers of 400 lb. pressure. The crude gives a yield of 12–14% of kerosine which is acid and earth refined, 800 to 900 brls. being turned out daily. C. L. G.

241. No Condensers Used in Topping Unit. W. T. Ziegenhain. *Oil and Gas J.*, 1930, **29** (31), 30, 107.

The Crew Levick Co. are operating two topping units designed by D. G. Brandt, of the Doherty Research Co., which are of unique design, in that no condensers are included, this work being done by heat exchange with incoming crude. The capacity of each unit is 2500 brls. crude, taking 50% overhead, although at present the plant is handling 3250 brls. Venezuelan crude with 30% overhead.

Fractionation is effected by the heated vapours passing through a series of exchangers counter current to the incoming crude. The plant consists of a tower with 6 bubble trays set at an angle, six tower exchangers, and 4 vapour exchangers. The furnace required is very small, only 1800 feet of 4 in. tubing being required. The tower exchangers are of the tubular type (10 ft. by 3 ft.), but each is divided into sections. Through one of these the overhead vapours pass, through

another the hot fuel, and through the third the hot gas oil—the latter two making a return journey through the exchanger, so that the incoming crude receives heat from all the products made on the plant. Actually it is heated to 450° F. before entering the furnace and leaves it at only 660° F. Control is effected by allowing varying amounts of gas oil and fuel to run through all or one of the exchangers. The main advantage in fractionation is that the temperature gradient is constant in contrast to that in a normal tower. There is less danger of loading the tower owing to the fact that fractional condensation takes place in an area where the temperature difference between liquid and vapour is not great at any point.

When running on 18/18·5 gravity Venezuelan crude the following yields were obtained:—Gasoline (425° F. endpoint), 10%; Furnace oil (130° F. flash), 9%; Gas oil (27 gr.), 11·5%; Fuel oil (13 gr.), 68·0%. A flow sheet is given. C. L. G.

242. Perfect Flexible Method for Treating. C. O. Willson. *Oil and Gas J.*, 1930, **29** (31), 38.

The Union Oil Co. has perfected its continuous gasoline and special naphtha treating plant at Los Angeles Refinery, so that it is now largely automatic in action, only one man per shift being required. As a result the quantity of acid used has been considerably reduced and a 30 color stable product given. The treatment given consists of acid followed by clay contacting. The plant is very flexible and easily handles the different types of products produced by the refinery.

The most suitable quantity of acid is first determined in the laboratory and the necessary amount passed into the gasoline in a baffled mixing coil. The treated stock is then passed into the base of a packed sludge settling tower, where a large part of the sludge separates and is removed and eventually treated for recovery of acid. The stock passes to a second settling drum, which it leaves practically acid free and passes into the clay agitator. It is here thoroughly mixed with Death Valley 200-mesh clay to remove any trace of acid left in the gasoline and any other impurities. The clay gasoline mixtures then pass to a clarifier, where the gasoline is separated by passing under pressure through a bed of fine gravel and passed direct to storage. Paddles skim the sand-clay mixtures from the top of the bed; it is then passed into a clay tub where fresh water is added, and then pumped to a settling and skimming tank. Settlement is accelerated by acid and salt water, the slops containing a small percentage of gasoline being removed from the top of the tank and the gasoline recovered later. The clay is removed from the bottom of the tank to a dump.

An interesting feature of the Los Angeles plant is the use of hot gas oil from the Cross cracking plant to provide the heat for re-running the pressure distillate, the gas oil not being recycled owing to its having a favourable commercial value. C. L. G.

243. Operating Largest Kerosine Plant. C. O. Willson. *Oil and Gas J.*, 1930, **29** (32), 38, 131.

A 900-ton Edeleanu unit, capable of treating 7,000 barrels a day of kerosine distillate, is now in operation at the Los Angeles refinery of the Union Oil Co. It is believed to be the largest single plant in the U.S.A., and cost approximately a million dollars.

The chemical composition of California kerosine distillate is such that the cost of refining to the required quality with acid is often prohibitive. In this plant the distillate is passed through a tower packed with rock salt to remove moisture, and is then de-aerated under partial vacuum. After cooling to 15° F. the distillate is treated with an equal volume of liquid SO_2 in a vertical mixer. The treated kerosine, containing about 10% of SO_2, leaves from the top, and the extract, together with the remainder of the SO_2, from the base of the mixer. The SO_2 is removed from both products in the usual way, a temperature of 150° F. being employed for the kerosine and 170° F. for the extract. Increasing stages of vacuum are used on the second and third evaporators, and over 95% of the SO_2 is recovered. Throughout the plant the maximum use is made of heat and cold exchangers.

No further treatment is given to the kerosine, which meets all the requirements of the export and domestic markets. The quantity of extract obtained is equivalent to 15% of the intake of kerosine distillate. The article contains a flow sheet and information concerning the accessory plant. R. A. E.

244. Treating Equipment Reduces Costs. C. O. Willson. *Oil and Gas J.*, 1931, **29** (33), 25, 104.

The Rio Grande Oil Co. have effected considerable economies in treating operations at their Vinvale (Calif.) refinery by the installation of separate equipment for treating straight-run gasoline, pressure distillate and cracked gasoline. This refinery has a capacity of 10,000 brls. daily and possesses two Jenkins cracking units. The crude is chiefly Elwood and Santa Fe Springs.

The straight-run gasoline is treated with 66° Bé. acid, and discharged through a permeable filter bed composed of a material not attacked by acid and which, owing to the difference in viscosity between the acid and the gasoline, maintains a different rate of flow in each fluid, thus resulting in fresh contact of the acid with the gasoline. It also helps to coalesce the acid particles, accelerating separation. The acid gasoline flows through a horizontal desludger and is contacted with a dry neutralization agent, then to a settling tank, and finally to another contacting plant with 200-mesh clay, the clay being finally removed by Cannon filters.

Pressure distillate is first contacted with caustic soda solution and then contacted with acid in the same type of treater used for straight-run gasoline. It is then similarly desludged and contacted with caustic soda to remove any acid present. The main feature of the Cannon desludgers and contactors is the mechanical separation of the sludge, eliminating the necessity of water washing the acid gasoline, which usually results in the re-solution in the gasoline of a certain amount of the sludge.

Cracked gasoline is first mixed with sulphur oil (cracked gasoline filtered through sulphur) and contacted with plumbite in two Cannon contactors and filters.

The resulting straight-run and cracked gasolines are W.W., sweet and have negative doctor tests, the sulphur content of the straight-run being 0·05% and that of the cracked 0·08%. The cost of the chemicals used in (a) straight run, (b) pressure distillate and (c) cracked gasoline

is given as (a) $0·0098, (b) $0·055, (c) $0·005 per brl., and the amount of chemicals; (a) 0·2 lb. acid, 0·1 lb. neut. reagent, 0·1 lb. clay; (b) 2½ lb. acid, 0·25 lb. caustic soda; (c) not given.

Flow sheets of the three treating plants are given. C. L. G.

245. Conveyor Simplifies Handling of Coke. C. H. Wilson. *Oil and Gas J.*, 1930, **29** (28), 34, 104.

A power operated conveyor for handling a coke output of 150 tons daily has been in operation for the last three years, in a refinery in Wyoming. The main features of the conveyor are as follows.

The conveyor operates in a concrete tunnel which is located below the concrete supports over which the reaction chambers are located.

The endless conveyor is the steel apron type supported by structural steel in the tunnel. At one end the horizontal conveyor connects to an inclined conveyor which empties into a coke hopper. Both conveyors are able to handle powdered as well as lump coke. The coke is removed from the reaction chambers by means of a cable with hoist, the cable being inserted in the chamber before the cracking operations start. The power is transmitted by means of a 25 h.p. steam engine.

High pressure lubrication has been installed and this has completely overcome the trouble often experienced in conveyor systems of this type.

Practically all manual handling of the coke has been eliminated with the installation of this conveyor. L. W. C.

246. Flue Gas for Reducing Fire Hazard. A. E. Pew. *Refiner*, 1930, **9** (12), 122.

In connection with the elimination of potential causes of fire in oil refineries, the problem of preventing the presence of explosive mixtures is discussed and three methods for its solution suggested. The first consists of the use of gas as an enrichment agent and as the displacement medium in tank breathing, thus eliminating the presence of air. The second and least used method comprises the forced circulation of air through tanks containing small amounts of dissolved gases, thereby preventing the tank atmosphere exceeding the lower explosive limit. The third and most widely used method is that of utilizing flue gas to purge gas or oil vapours from various types of refinery equipment, including tank ships and barges. The hazards eliminated are the danger of discharge from static and bound charges, the spontaneous ignition of pyrophoric iron and sulphur corrosion compounds, all being ignition agents in the presence of explosive atmospheres. The flue gas system makes equipment safe for repair and inspection without the use of steam. W. H. T.

247. Cleaning Devices for Heat Exchangers. C. A. Hubert. E.P. 339,806, 21.12.29.

This relates particularly to heat exchangers consisting of ribbed tubes in which the accummulation of impurities, especially soot, is unavoidable, such as air heaters heated with waste gas, feed water heaters, etc.

Stripping means such as brushes, scrapers, etc., are driven through the waste gas ducts and are shaped so that their driving requires

relatively little power. During their passage through the smoke flues the section of free passage of the flue is not essentially reduced.

The brushes are drawn through the flue sections on endless chains which pass over pulleys mounted on sleeves. These sleeves are carried on rotary shafts extending parallel to the longitudinal axis of the tubes in the heater, and a suitable drive is provided for imparting rotary movement to the upper shaft. W. L.

248. Alkaline Treatment of Petroleum Vapours. R. C. Wheeler and P. W. Prutzman. U.S.P., 1,784,262, 3.12.30. Appl., 24.6.27.

Various fractions of petroleums containing phenols and naphthenic acids are purified by the conversion of such bodies into salts and soaps. Further, such reaction products are withdrawn from the vapours completely and continuously and without the production of emulsions or the use of wash water. A mixture of vapours and steam is contacted with a solution of sodium hydrate without substantial condensation of the petroleum, thereby producing a reaction product insoluble in the solution. The latter is maintained in continuous contact with the vapour mixture and at such a concentration as not to evolve or absorb a material quantity of steam during contact. Claims are made for the apparatus. W. H. T.

Cracking.

249. Corona Discharge as Accelerator in Vapour phase Cracking H. R. Rowland. *Oil and Gas J.*, 1930, **29** (28), 38, 92, 95, 97.

The chief difficulty encountered in the use of the corona discharge in connection with vapour phase cracking was the provision of an insulator of sufficiently high dielectric strength when working at a temperature of 1000° F. which would withstand the mechanical distortion, due to the fact that the lower part of the insulator was exposed to the hot gases, while the upper part was at atmospheric temperature and separated by a narrow middle zone in the packing gland, through which the temperature gradient was very steep. Experiment showed that fused Brazilian rock quartz could be utilized, as although the dielectric strength diminished with rise of temperature, it was mechanically perfect, in that the coefficient of thermal expansion was practically zero. An experimental plant has been constructed, and has been running successfully.

The charging stock is pumped under pressure through a pipe still into a vaporizer in which about 1½ lb. pressure is maintained, and into which a current of hot methane is passed. This serves three useful purposes. 1. It reduces the vapour pressure and gives better vaporization. 2. It acts as a noncondensible carrier gas assisting the vapour flow through the system. 3. Fixed gases made in the cracking operation and recycled with methane apparently have a hydrogenating effect in the cracking zone, preventing formation of carbon as well as lowering the gravity of the recycling stock.

After passing through a preheater the vapours enter the reaction tubes which are kept at cracking temperature.

The reaction tubes are 15 ft. lengths of 12in. pipe, set vertically and exactly in the centre of the top of each tube is placed the insulator from which hangs a small diameter wire of nichrome steel. The wire has an insulating bob suspended on the end and slides in an insulating thimble, thus permitting expansion of the wire, but prohibiting any lateral movement.

After being acted on by this silent corona discharge, the vapours pass to the usual fractionating column, condenser, and gas separator.

Two theories are advanced as to the actual effect of the corona discharge. 1. That by more perfect ionization of the vapours than heat alone can effect change in the molecule can more readily take place. 2. That the discharge, acting as an agitator, keeps the vapours in a state of high turbulence, driving them against the heated walls of the tube, thus assisting the cracking process.

When the plant was operated without the discharge the percentage make of spirit was lower, as was the antiknock value of the finished gasoline.

The usual methods of refining by acid, plumbite, etc., were found to be unsatisfactory, so a new method of treatment was devised of which only an outline is given. By this means a completely satisfactory and stable spirit was produced. H. B. T.

250. Treatment of Cracked Gasoline with Surakhani Clays. E. Ginsburg and A. Markariants. *Azerbaijan Oil Ind.*, 1930, No. 10, 93–98.

Greyish-green clays from lower strata were used for the experiments. The clay contained carbonates, and had an alkaline reaction. Fine clay powder heated up to 120° C. gave the best results. Clay activated by acids was unsatisfactory. The clay treatment alone was insufficient. Aromatic and unsaturated compounds, resins and sulphur compounds were not affected, or affected only slightly by the treatment. Additional treatment with 0·1 to 0·5% of H_2SO_4, sp. gr. 1·84, gave very satisfactory results. It is intended to repeat the experiments on an industrial scale. P. A.

251. Obtaining Light Hydrocarbons by the Action of Nascent Hydrogen on Carbonaceous Material. J. Fohlen. E.P. 338,544. 21.6.29.

Light hydrocarbons are produced from carbonaceous materials containing heavier hydrocarbons capable of decomposition by cracking without hydrogenation. The affinity of the organic bonds released is utilized to connect thereto active atoms of nascent hydrogen.

W. H. T.

252. Conversion of Hydrocarbons into Those of Lower Molecular Weight. J. Y. Johnson, Assr. to J. G. Farbenind. A.-G. E.P. 339,274. 30.5.30.

Claims are made for the conversion of hydrocarbons by cracking in the vapour phase. The initial material is subjected to a high temperature in the presence of a catalyst consisting of one or more pieces of free elementary silicon or the latter deposited on a carrier.

W. H. T.

253. Cracking of Hydrocarbon Oils. W. W. Triggs, Assr. to Panhandle Refining Co. E.P. 339,291. 29.6.29.

The process comprises preheating the oil, introducing it into a vaporising zone and then passing the vapours through a superheated cracking zone. Make up oil is introduced into the system and heated by indirect contact and in counter-current with the vapours passing from the cracking zone through a plurality of heat exchanging and fractionating zones. W. H. T.

254. Converting Petroleum Oils. Petroleum Conversion Corpn. E.P. 340,021. 19.9.29

A system of vapour phase cracking is described in which oil vapour is mixed with a heated carrier gas of such a quantity and temperature as to bring about the conversion reaction. The oil to be converted is fractionally vaporized and the fractions are introduced into the reaction zone at separate points along the path of the reacting products, the lighter fractions being subjected to a longer time of conversion. The carrier gas is also added at a plurality of separate points along the path of reaction, the temperature being thus controlled as desired. The carrier gas is preferably introduced at the same points as the various oil vapour fractions. W. H. T.

255. Cracking Hydrocarbon Oils. J. E. Pollak, Assr. to Petroleum Conversion Corpn. E.P. 340,030. 19.9.29.

This process is substantially the same as that described in the previous abstract, but more operating details are given. The amount of carrier gas added in each successive stage is sufficient to raise the temperature to that of initial conversion, and thus a constant mean temperature is maintained throughout the operation. The exit temperature of the last stage is not more than 100° below the cracking temperature. Three stages are provided, one-half of the carrier gas is added in the first stage, and a quarter at each of the successive stages. W. H. T.

256. Cracking Hydrocarbon Oils. J. G. Hawthorne, Assr. to W. M. Cross. U.S.P. 1,782,686, 25.11.30. Appl., 9.7.27.

The process described claims, firstly the superheating of the hydrocarbon vapours after they have been raised to vaporising temperatures, whereby a major part of the cracking is brought about in the vapour phase, secondly the cooling of the cracked products subsequent to the superheating stage in order to separate the heavier from the lighter fractions, and thirdly the automatic balancing of pressure between the vapours passing through the superheater stage and the flue gases supplying heat to these vapours thus admitting of the use of special highly refractory material for the tubes through which the vapours pass. The cracking stock is pumped through a pipe still consisting of radiant and convection banks of tubes and leaves at a temperature of 800 to 1000° F. and at any pressure up to 1000 lbs./sq. in.

The pressure is then allowed to fall to atmospheric and steam is introduced into the system. The vapours pass through the superheater, which may be composed of tubes of carborundum, alundum or ceramic fire-

resisting materials. The superheater furnace is top fired and the damper is automatically controlled to maintain the same pressure in the furnace as obtains within the superheater tubes. The vapours leave the superheater at 900° to 1200° F. and are then cooled to about 750° F. in a heat exchanger and pass to a separator, the level of condensed liquid in this being automatically kept constant. From the separator the lighter vapours are taken to a bubble cap dephlegmating column; through a coil in the top of this the charging stock is pumped to provide reflux, and the condensed liquid from the bottom of the tower, again automatically controlled, is returned to the charging lines. H. B. T.

257. Cracking Hydrocarbon Oils. R. J. Dearborn, Assr. to Texas Co. U.S.P. 1,783,010, 25.11.30. Appl., 19.1.28.

The plant described consists of two interconnected units, one a liquid phase and the other a vapour phase cracking system. Charging stock is pumped through a pipe still and enters a soaking drum at a temperature of 750° to 950° F., and at a pressure of one hundred to several hundred lbs./sq. in. Through a coil suspended in this drum are passed the vapours from another pipe still at a temperature of 1000 to 1200° F. whereby the oil in the soaking drum is maintained at any desired cracking temperature.

The vapours from the drum pass to a separator and a condenser, heavy residue being drawn from the bottom of the drum.

The vapours from the coil pass into a tar leg and thence to a separator and a condenser.

The top and bottom of the soaking drum are removable for cleaning purposes. H. B. T.

258. Process for Cracking Petroleum Oil. C. P. Dubbs. U.S.P. 1,783,228, 2.12.30. Appl., 8.10.20.

The oil to be cracked is passed through a series of stills of successively increasing temperature, and vapours are taken off from each still and passed to a reflux condenser individual to each still. The reflux condensate separated from the vapours removed from a still of lower temperature is returned directly to a still of higher temperature. The vapours which are not condensed in the reflux condenser are passed to a still of lower temperature. Finally vapours are taken off from the reflux condenser connected to the still of lower temperature. H. E. T.

259. Process and Apparatus for Treating Oils. C. P. Dubbs. U.S.P. 1,783,229, 2.12.30. Appl., 14.9.28.

The apparatus consists of a furnace in which is seated the still, which may take the form of a vertical chamber, having a partition forming a narrow shelf or chamber which is spaced away from the wall and bottom of the still. The upper end of the shell is provided with a flange extending to the outer wall of the still so as to seal the upper end of the passage leading from the still. Oil is permitted to pass out of this passage through a plurality of pipes open at their lower ends, which may be removed for cleaning when necessary. The vapour space of the still is provided with a vapour outlet pipe having a throttle valve and leading

to the side of the dephlegmator, which is provided with baffles. The upper end of the dephlegmator is connected by a pipe having a throttle valve to a condenser coil seated in a condenser box, the lower end of the coil being connected by a pipe to the top of the receiver. The receiver is provided with a pressure gauge, a liquid level gauge and a gas outlet pipe. Raw oil may be fed to the top of the dephlegmator through a feed pipe, where it is preheated, mixed with the reflux condensate and returned to the lower end of the passage leading from the still. The process may be operated at any desired temperature and pressure, *e.g.*, the oil may be heated to a temperature of 800° F. and subjected to a vapour pressure in the still and condensing apparatus of from 75 to several hundred lbs. H. E. T.

260. Converting Petroleum Oil. G. Egloff and J. C. Morrell, Assr. to Universal Oil Products Co., Chicago. U.S.P. 1,783, 231. 2.12.30. Appl. 1.12.22. Renewed 5.2.29.

The plant follows accepted design for liquid phase cracking, and consists of tube still, expansion chamber, dephlegmator, condenser, receiver, and gas separator. From the two latter gas lines lead to the bottoms of a number of vertical cylindrical vessels. These vessels have a zone of chequer brick work in the lower part, and above this they are filled with broken adsorbent material such as charcoal. The gases from the cracking plant are passed upwards through one or more of these vessels until the adsorbent material is saturated with hydrocarbons.

The gas stream is then passed to other vessels, and heated flue gases are used to free the material of its adsorbed hydrocarbons. Steam may be introduced at the same time with advantage in many cases.

H. B. T.

261. Process and Apparatus for Converting Hydrocarbons. J. C. Morrell and H. P. Benner. U.S.P. 1,783,257. 2.12.30. Appl. 17.7.24.

A maximum production of lighter hydrocarbons is obtained from heavy hydrocarbons by subjecting the latter to the action of heat under superatmospheric pressure in order to cause vaporization, then withdrawing the vapours and subjecting them to reflux condensation. The unvaporized residue is pumped to a coking still, wherein the pressure is substantially reduced, thus causing the lighter fractions to be released due to their latent heat. The lighter fractions and residue from the coking still are withdrawn and separated. The reflux condensate from the primary cracking operation may be returned and mixed with the residue being withdrawn from the primary cracking operation immediately before being acted upon by the pump for the purpose of agitating and maintaining the residue in a turbulent condition. By this means free carbon particles produced during the cracking operation are held in suspension in the residue and withdrawn in that condition into the coking still. The reflux condensate obtained from the vapours obtained from the unvaporized residue in the primary cracking operation is injected without passage through the heating zone, back into the unvaporized oil in the reaction zone in order to maintain the unvaporised oil in a swirling motion, and to flush the walls of the reaction chamber and so prevent the formation of a hard deposit of coke. H. E. T.

262. Process for Cracking Petroleum Oil. R. T. Pollock. U.S.P. 1,783,259. 14.11.29.

The oil to be treated is subjected to a cracking temperature and pressure in a pressure zone. The high boiling fractions of the evolved vapours are separated from the low boiling fractions in the form of a condensate, and the condensate, while still hot, is subjected to further cracking temperature and pressure, while simultaneously distilling another independent body of oil. The vapours are separated according to their boiling points, and the condensate is co-mingled with the oil in one of the several pressure zones. H. E. T.

263. Cracking Oil. W. Brink and G. L. Hess, Assrs. to General Oil Products Co. of Delaware. U.S.P. 1,783,697. 2.12.30. Appl., 9.11.27.

The plant described is adapted to the cracking of heavy topped oils, and eliminates the use of any recycling pumps.

It consists of a well lagged vertical still 5 ft. in diameter and 32 ft. in height, provided with a gauge glass two-thirds of the way up. The still is connected at the bottom with a horizontal cycling drum, which is about the same length as the still, but only about three-quarters of its diameter. This drum is fitted with a number of vertical risers, each of half the cross sectional area of the drum, and from the top of each riser leads a 4 in. pipe. The pipes, nested together, pass at an angle of 45° upwards through a furnace and back again into the flue at an angle of 45° with the horizontal. The right-angled bends of these tubes are outside the furnace chamber for easy access for cleaning. The pipes then enter the still at a point 4 ft. above the oil level.

The top of the still is connected with a horizontal fractionating drum, and this in turn is connected at each end with another superimposed fractionating drum, from the top of which cracked vapours are taken to a condensing system.

Beneath and connected with the cycling drum are two carbon tanks in which the coke formed during the cracking settles out, and may be removed from the system.

The charging stock, preferably a heavy topped crude, is fed at a temperature of 400° F. to the bottom of the still, which is maintained at a temperature of 800°–900° F. under a pressure of 100–200 lb./sq. in.

The furnace is so designed that there is no possibility of flame playing on the circulating tubes.

If required, a fuel oil may be withdrawn from the cycling drum, but it is generally preferred to crack the oil down to gasoline and carbon. With heavy oils it is possible to recover up to 90% of pressure distillate. H. B. T.

264. Cracking Hydrocarbons. E. W. Beardsley and M. W. Colony, Assrs. to Petroleum Conversion Corpn. U.S.P. 1,784,126, 28.6.27.

The patent describes a process of converting hydrocarbon oils by storing heat in a carrier gas. The heated gas is mixed with the vapour of the product to be converted, the products of conversion being passed

from the reaction chamber through a heat exchanger, where the incoming hydrocarbon material is vaporized. The vapour produced is passed in heat exchange relationship with the products of reaction and thence into the reaction chamber. The heat carrying gas entering the cycle is passed through another heat exchanger to a gas heating device and thence to the reaction chamber. W. H. T.

265. Apparatus for Cracking Hydrocarbons. W. Miller, Assr. to Continental Oil Co., Oklahoma. U.S.P. 1,785,042, 16.12.30. Appl., 8.6.27.

A cracking element is maintained at a cracking temperature and the vapours are passed to a dephlegmator which consists of a refluxing zone where the high-boiling fraction is condensed, and a relatively cool bubbling zone provided with pools of hydrocarbon liquid through which the vapours are discharged to the refluxing zone. A heat exchanger is inserted beyond the bubbling zone to receive vapours passing from this zone and a final condenser is placed beyond the exchanger. Means are provided to pass the charging stock through the heat exchanger into the refluxing zone without entering into the liquid in the bubbling zone, also means for transmitting the mixture of charging stock and reflux condensate from the refluxing zone to the cracking element. A conductor discharges the condensate from the bubbling zone to a storage receptacle. W. S. C.

266. Treating Petroleum Oils. W. M. Cross, Assr. to Gasoline Products Co. U.S.P. 1,785,073, 16.12.30. Appl., 28.8.25.

The oil is heated in a coil and passed to an enlarged chamber in which it is maintained at a conversion temperature and pressure in excess of 450 lbs./sq. in. to retain it substantially liquid. Sufficient quantities of a gas containing free oxygen are introduced at several points along the bottom of this chamber so as to maintain the thermal conditions of conversion and to increase the formation of antidetonating compounds. The pressure on the oil from the chamber is reduced in a vaporizing stage and a distillate obtained by condensing the evolved vapours. W. S. C.

267. Art of Pyrolysis. N. E. Loomis, A. H. Tomlinson and F. A. Howard, Assrs. to Standard Oil Development Co. U.S.P. 1,785,274, 16.12.30. Appl., 31.5.23.

An apparatus and process is described for the pyrolitic conversion of hydrocarbons into lower-boiling products. A stream of the hydrocarbons is heated in a heating zone and undergoes cracking, which is allowed to continue in a digestion zone, apart from the heating zone which is connected by means of a passage to the digestion zone. A feed pipe is fitted to the heating conduit and means are available for supplying relatively cool oil to the digesting passageway as desired so that the temperature in the digestion zone is lower than that in the heating zone, but not below an effective cracking temperature. A by-pass pipe leads from the heating conduit around the passageway and leads directly to the rectifying tower. W. S. C.

See also Abstract No. 176.

Hydrogenation.

268. Destructive Hydrogenation of Carbonaceous Liquids. W. R. Tate, H. P. Stephenson and Imperial Chemical Industries, Ltd. E.P. 338,150, 13.8.29.

By carbonaceous liquids is meant oils or suspensions of coal in oil or the like. The process is conducted in the liquid phase and includes the steps of reacting upon the raw material with hydrogen in a reaction vessel inside a high pressure container and separating the liquid and gaseous products at a temperature lower than the reaction temperature in a vessel situated in the same high pressure container. The reaction products may be reduced in temperature by heat exchange with incoming hydrogen or with the incoming liquid material, before passing into the separating chamber, and the heavier products condensed whilst the light oils remain in the vapour state and are withdrawn.

The heavier product passes out, in heat exchange with the raw material, and after separation of solid matter is returned for further treatment.

Two forms of the invention are described. R. P.

269. Liquid Products from Coal or other Carbonaceous Material. N.V. de Bataafsche Petr. Mij. E.P. 338,192, 14.8.29.

Treatment of carbonaceous material with hydrogen at elevated temperature and high pressure converts the raw material into lighter products and to facilitate the reaction catalysts are used. It has been found that molybdenum compounds when applied in a special form have an enhanced effect and yields are greater. The catalyst consists of a mass of finely divided decolorizing carbon, or other absorbing substance, intimately mixed with finely divided molybdenum or compound thereof. The molybdenum or its compound should be in a colloidal state and absorbed by the carbon.

The preparation of the catalysts is as follows—15 g. ammonium molybdate are dissolved in 400 c.c. of water and 100 c.c. of 3 to 4 normal H_2SO_4 is added. The solution is heated nearly to boiling then reduced with H_2S whereby a blue colloidal solution is formed. To this solution 40 g. "carboraffin" are added which absorb the colloidal matter and decolorize the solution. The carbon is filtered, washed with a small quantity of water, and dried, first at ordinary temperature, then at 50° to 60° C. Using the prepared catalyst 16% of product boiling below 300° C. was formed whilst under the same conditions, but using molybdic oxide as catalyst, only 9% of product was produced. R. P.

270. Destructive Hydrogenation. H. P. Dean and Imperial Chemical Industries, Ltd. E.P. 339,040, 4.10.29.

It has previously been proposed to pass the hydrogen through a heating jacket surrounding the liquid material in the reaction vessel.

In the present invention the hydrogen is passed through a heat exchanger immersed in and surrounded by the material in the reaction chamber. Thus the area of indirect contact between the hydrogen before its entry into the reaction zone and the reaction liquid is increased to secure more effective heat exchange. This may be accomplished by providing a plurality of tubes in parallel or by lengthening the path of the

hydrogen by using a coil situated within the reaction chamber. Thus in the case of an endothermic reaction heat is supplied by the preheated hydrogen and the heat exchanger promotes a heat exchange from the hydrogen to the liquid in the reaction zone In the case of an exothermic reaction, the hydrogen, at a lower temperature, abstracts heat from the reaction zone and maintains the temperature at the desired point.

R. P.

271. Treatment of Carbonaceous Materials. F. Uhde. E.P. 339,317, 5.9.29. Conv. (Germany), 3.10.28.

Without the use of compressed hydrogen or special compression pumps hydrogenation of carbonaceous materials may be effected by the action of spongy iron and water in pressure tight vessels.

It is claimed that pure hydrogen and its compression are dispensed with; the hydrogen is more active, as it is in a nascent state; and a mixture of iron and water does not attack the walls of the reaction vessel.

Different examples are given, such as:—Three parts of topped petroleum are heated with one part of spongy iron and one part water in an autoclave to between 400 and 470° C., the pressure rising to 200 atm. Apart from gas, which is chiefly hydrogen, 60 to 80% of the product boils below 200° C. and 10 to 20% below 360° C. The heavier portions can be recycled.

The gas may be used for the regeneration of the oxide of iron formed.

R. P.

272. Destructive Hydrogenation of Carbonaceous Materials. W. R. Tate, H. P. Stephenson, J. F. Lehmann and Imperial Chemical Industries, Ltd. E.P. 339,479, 29.11.29.

The invention relates to a liquid level indicator in conjunction with a vertical reaction chamber surrounded by a vessel which acts as a separator for the liquid and gaseous products. The feed enters the central vessel at the bottom, slightly above the hydrogen inlet, and the liquid products overflow into the annular space near the top of the chamber. The liquid product is withdrawn at the bottom of the annular space and the gases pass out at the top of the chamber.

Two tubes enter the gas space of the reaction vessel, one is open to the gas, whilst the other extends down through the liquid to the bottom of the annular space. These pipes are connected through catchpots to the ends of a U tube gauge. The gauge is calibrated to indicate the level in the annulus, and means are provided to ensure that the tubes are gas filled. The feed or outlet may be controlled, according to the registered level on the gauge to ensure steady conditions. R. P.

See also Abstracts Nos. 317, 318, 323, 325.

Chemistry of Petroleum.

273. Alkali Organic Experiments. E. Bergmann. *Ber.*, 1930, **63** [B], 2593–2597.

The transformation of $\alpha\alpha\zeta\zeta$ tetraphenyl-$\Delta^{\alpha\zeta}$-hexadiene into a potassium derivative which yields $\alpha\alpha$-diphenyl-Δ^{α}-propene and $\beta\beta$-diphenylacrylic acid is explained by the hypothesis that addition of the metal occurs initially at the $\alpha\zeta$ carbon atoms and the free valencies of the $\beta\xi$

carbon atoms saturate themselves in pairs by the formation of the cyclobutane derivative, $CHR\langle{}^{CHR}_{CH_2}\rangle CH_2$ ($R=C(C_6H_5)_2K$). By reason of ring tension the latter compound is unstable and yields two molecules of potassium diphenylvinyl. The following experimental evidence is cited.

Methyl suberate, prepared quantitatively by the action of diazomethane on the acid, is converted by magnesium phenyl bromide into $\alpha\alpha'\theta\theta'$-tetraphenyloctane-$\alpha\theta$-diol, and thence by boiling with glacial acetic acid containing a little sulphuric acid, into $\alpha\alpha'\theta\theta'$ tetraphenyl-$\Delta^{\alpha\alpha'}$-octadiene, melting at 92–93°. The hydrocarbon gives a lithium derivative converted into a hydrocarbon, $C_{32}H_{32}$, of melting point 212–213°, which is regarded as 1:2 dibenzhydrylcyclohexane since it is readily dehydrogenated by treatment with sulphur; the freedom from strain of the cyclohexane system renders a secondary reaction unnecessary. The instability of the cyclo-butane system leads to an analogous secondary decomposition during the withdrawal of metal from compounds of the type disodium tetraphenylbutyl,

$$(\cdot CH_2\cdot C\,Na\,Ph_2)_2 \longrightarrow CH_2\langle{}^{CPh_2}_{CH_2}\rangle CPh_2 \longrightarrow 2CH_2=CPh_2$$

The presence of the butane skeleton in this compound, previously obtained only by the addition of sodium to as-diphenylethylene, is established by the observation that it is obtained by the action of sodium on $\alpha\delta$-dimethoxy-$\alpha\alpha'\delta\delta'$-tetraphenylbutane of m.p. 230° after softening at 227°, prepared by the action of sulphuric acid on tetraphenylbutanediol in a mixture of methyl alcohol and dioxan. Treatment of the butanediol with potassium and methyl iodide in boiling xylene yields the corresponding monomethyls ether. $\alpha\alpha'\delta\delta'$-tetraphenyl-$\Delta^{\beta}$-butinene-$\alpha\delta$-diol is transformed by sulphuric acid in methyl alcohol at ordinary temperatures into the dimethyl ether which is hydrogenated in boiling propyl alcohol in the presence of palladised barium sulphate to α-methoxy-$\alpha\alpha'\delta\delta'$-tetraphenyl-n-butane of m.p. 140–141°. Treatment of the dimethyl ether with sodium followed by hydrolysis gives two hydrocarbons of m.p. 97° and 167° respectively, whilst the sodium derivative with phenylthiocarbimide yields the dithioanilide of $\alpha\alpha'\delta\delta'$ tetraphenyl-$\Delta\beta$-butinene-$\alpha\delta$-dicarboxylic acid of m.p. 204°.

W. S. C.

274. Studies on Polymerisation and Ring Formation: VII. Normal Paraffin Hydrocarbons of High Molecular Weight Prepared by the Action of Sodium on Decamethylene Bromide. W. H. Carothers, J. W. Hill, J. E. Kirby and R. A. Jacobson. *J. Amer. Chem. Soc.*, 1930, **52**, 5279–5288.

Hydrocarbons of the general formula $H[(CH_2)_{10}]_xH$ have been prepared by the action of sodium on decamethylene bromide in ethereal solution. The normal course of this reaction, including that due to participation of the solvent, is to give norml paraffins of very high molecular weight. A mechanism of the reaction is suggested and the absence of cyclic hydrocarbons explained. It appeared to be impossible to prepare paraffins higher than C_{120} by this method.

The hydrocarbon mixture obtained was separated by the employment of a molecular still in which the pressure was continually maintained below 10^{-5} mm. Fractions were collected between 130° and 300° C. with corresponding melting points ranging from 35° to 106·5° C. 50% of the initial mixture was undistillable. The rate of distillation was very slow—each fraction took about 20hr.—but increased on redistillation of the partially purified materials. A table of the melting points and distillation temperatures is given.

In order to identify the hydrocarbons obtained, comparison was made with known paraffins. The melting points, 36·7° and 65·6–66° respectively, have been assigned to *n*-eicosane and *n*-triacontane both in good agreement with the observed values. The other four hydrocarbons are new and the last stands six C atoms above any paraffin previously described. From available data of intermediate members of the same series as those under consideration it has been found possible to construct a smooth curve of melting points covering the range C_{19}–C_{70}. There has been some doubt as to the constitution of dimyricyl, but the present data confirm the conclusion that the hydrocarbon is $C_{60}H_{124}$ as the alternative formula $C_{30}H_{122}$, hexacontane, has m.p. 98·5–99·3° as here recorded.

From the distillation temperatures, it has been found possible to make a rough estimate of the order of magnitude of the vapour pressures of these hydrocarbons. A value of less than 0·01 mm. for heptacontane at 300° and a value less than 1 mm. for triacontane at 100° are indicated from these calculations. Heptacontane has a molecular weight of 983, and it is probable that no organic compound of much higher molecular weight than this can be distilled under any conditions, however favourable. This is confirmed by comparison of the values of the molecular cohesion of heptacontane and of octacontaine with the heat of separation of the carbon-carbon bond.

The residue from the distillation undoubtedly contained *n*-octacontane, *n*-nonacontane, *n*-decacontane and higher hydrocarbons and had an apparent average molecular weight of about 1300. It was separated into fractions melting at 100–107°, 106–111° and 110–114° respectively.

When samples of the hydrocarbon from C_{30} to C_{70} were heated at 410° in sealed tubes for varying periods, it was found that the decomposition or cracking—measured by lowering of melting point and evolution of gas—increased with increase of molecular weight. In fact, the decrease in the thermal stability with increasing molecular weight which is so marked in passing from methane to ethane and from ethane to hexadecane is still detectable in going from triacontane to heptacontane. However, it has fallen to such a small value that it is unsafe to infer that a hydrocarbon of molecular weight 200,000 or more might not persist at room temperature. W. D. S.

275. Partial Combustion of Methane at Various Pressures with particular Consideration of the Formation of Acetylene. F. Fischer and H. Pichler. *Brennstoff-Chem.*, 1930, 11, 501–507.

Experiments are described in which methane is passed at various pressures through capillaries heated to 1000–1600°. The gas with known streaming velocity is mixed with air or oxygen similarly heated of known streaming velocity before entering the oven containing the reaction zone. An apparatus is fitted to the oven for the separation of

water and subsequently of light oil and tar; for lower pressures a pump is placed between this apparatus and the gas meter. A special bomb was used for the explosion experiments.

The results show that in the partial combustion of methane the time and temperature are important factors. Primarily CH and OH radicles are formed, secondarily these are converted into carbon monoxide and hydrogen, the reaction being accelerated by catalysts.

The greatest yields of acetylene are obtained when the reacting gases are subjected to a short heating period. The gases are led relatively slowly through small disc-shaped spaces and then passed through a long, narrow tube with a greater velocity The yields of acetylene can amount, in the given temperature range, to 50–60% of the initial methane. The remaining carbon from the methane and part of the hydrogen are burnt to form carbon monoxide or water; this reaction provides heat for the formation of acetylene.

If a mixture of carbon monoxide and hydrogen is desired instead of a maximum yield of acetylene, lower temperatures can be applied and the conversions are not so much influenced by the heating period.

Diminished pressures give no essential increase in the yield of acetylene, but show a slower secondary combustion of acetylene. On the other hand, in the explosion experiments at high pressures greater quantities of the products of incomplete combustion are not detected.

Five and a half cu. m. of methane and 3 cu. m. of oxygen yield 1 cu. m. of acetylene and 8·5 cu. m. of a water gas rich in hydrogen such as is formed from carbon monoxide and hydrogen combined in the proportion of 1: 2. The acetylene can be separated from the cracked gas and used as a valuable initial product in organic syntheses. The mixture of acetylene and cracked gas can be used directly by the addition of catalysts in the production of hydrocarbons; $CO:H_2$ (1:2) is the best ratio for Fischer and Tropoch's synthesis of benzene. W. S. C.

276. A New Complex Hydrocarbon of the Probable Formula $C_{16}H_{14}$. C. S. Gibson and J. D. A. Johnson. *J.C.S.*, 1930, 131, 2785–2786.

During experiments on the synthesis of heterocyclic organic arsenical compounds from ββ′-dichlorodivinylchloroarsine $(CHCl=CH)_2AsCl$, a complex hydrocarbon was isolated by the following method.

The compound ββ′-dichlorodivinylchloroarsine was heated together with benzene and anhydrous aluminium chloride under a reflux on a water bath. A vigorous reaction ensued, much hydrogen chloride was evolved and a deep brown liquid remained. After 1¼ hrs. the product was cooled and ice added followed by hydrochloric acid and benzene. After thorough agitation, the liquid was filtered and the benzene layer separated, dried with calcium chloride and evaporated on the water-bath. The residue, distilled under reduced pressure, gave no unchanged ββ′-dichlorodivinylchloroarsine. The fraction boiling between 145° and 230°/9mm. was reddish-brown and readily solidified; it was readily soluble in alcohol when crude, but became sparingly soluble when pure. After two crystallisations the compound was obtained in pale yellow plates, melting at 179–180°, unchanged by further crystallisation. Its dilute alcoholic solution was pale yellow and exhibited a strong blue-violet fluorescence. Elementary analysis gave C 92·8, H 7·0, molecular weight in benzene 193, 198. Only oils could be obtained

from the alcoholic mother liquors. The hydrocarbon is stable to boiling alkaline permanganate, is moderately easily soluble in benzene and carbon tetrachloride when hot, but is sparingly soluble in the cold solvents. A dibromo derivative was isolated which points strongly to the formula $C_{16}H_{14}$ for the hydrocarbon. W. S. C.

277. Catalytic Reactions of Sulphur Compounds Present in Petroleum: I. High-Sulphur Naphthas in Contact with Nickel and Iron Catalysts. J. C. Elgin, G. H. Wilder, and H. S. Taylor.
II. Pure Sulphur Compounds in Hydrocarbon Materials in Contact with Nickel Catalysts. J. C. Elgin. *Ind. Eng. Chem.*, 1930, **22**, 1284–1293.

I. The preliminary results of an investigation carried out with the object of studying the reactions of sulphur compounds in petroleum in the presence of contact catalysts alone, or with addition of hydrogen, are described. Iron and nickel were chosen as catalysts and their effect in decreasing the sulphur content of two straight run and a cracked naphtha studied. Experiments were made in the vapour phase at 300° and 400° C. at atmospheric pressure, but determinations of the adsorption of sulphur from the naphthas in the liquid phase has also been studied. The A.S.T.M. lamp method was employed to determine the sulphur content and a negative doctor test was taken to indicate absence of mercaptans.

In the case of a sulphur-free nickel catalyst all sulphur could be removed from naphthas in the form of vapour. However, with continued use the catalyst became less efficient in its action, but it eventually reached a steady state, and then its effectiveness in reducing the sulphur content became constant. It was noted that fresh nickel catalyst was able to absorb sulphur from the naphthas in liquid form after they had been passed in vapour form over the catalyst. The addition of hydrogen increased the effectiveness of the catalyst in removing sulphur. The naphthas still showed differences in behaviour, but the characteristic action of the catalyst was the same. All mercaptans were removed, the product obtained throughout the run being sweet to doctor with each naphthas, an improvement on the result obtained without hydrogen.

Re-running the napthas over the catalyst at the same temperature, after it had reached the state where its action became constant, was studied. The results demonstrated conclusively that a nickel catalyst attains a state in which its action in removing sulphur becomes constant and does not tend to lose its effectiveness entirely with continued use. They also confirmed the conclusion that sulphur is removed when hydrogen is added to the naphtha vapour, which is not affected when it is not added. If the hydrogen was added to naphtha vapour passed in contact with the initially sulphur-free catalyst, a greater reduction in sulphur at the steady stage was effected than if the hydrogen was added after the catalyst had previously reached a steady state with naphtha vapour alone. Thus the action of hydrogen in increasing the effectiveness of the catalyst in removing sulphur is influenced by the previous history of the catalyst. It was also shown that hydrogen was able to remove sulphur from the surface of the catalyst.

The action of an iron catalyst showed a similar type of behaviour as nickel but was considerably less effective. It did not remove sulphur completely at any stage.

The removal of sulphur by adsorption from the naphthas in the liquid phase was determined. The amount of sulphur removed was proportional to the quantity of catalyst added until the sulphur had been reduced to a certain value. After this reduction very little adsorption took place. The adsorption was shown to be irreversible. The iron catalyst was a decidedly poorer adsorbent for the sulphur than nickel. All mercaptans were removed from the cracked naphtha, but not from the two straight-run naphthas. It was evident from the results that sulphur constituents are selectively adsorbed from naphthas.

Other properties of the naphthas besides sulphur content were appreciably altered. The odour was considerably improved in most cases and all colour could be removed from the naphthas in liquid form. In the vapour form with hydrogen most of the colour was removed. The A.S.T.M. distillation was not altered materially by passage over the catalyst in vapour form.

The preliminary results suggest that, with addition of hydrogen, alternate sulphide formation with the catalyst surface and its reduction by hydrogen may play an important *rôle* in the removal of sulphur, and, in consequence, that variation in the ratio of hydrogen pressure to the sulphur compound pressure may increase the effectiveness of the catalyst action.

II. Naphtha solutions of butyl mercaptan, isobutyl mercaptan, propyl sulphide, isobutyl sulphide, and thiophene, present individually, have been studied in contact with nickel catalysts. The experimental procedure was essentially the same as that employed in Part I. The data presented demonstrate differences in the reactivity of sulphur compounds. When no hydrogen was added, it was observed that both thiophene sulphur and butyl mercaptan sulphur were removed by reaction with the sulphur-free nickel surface, but to a widely different degree. The catalyst reached a state of constant activity in which it catalysed a reaction of butyl mercaptan which evolved hydrogen sulphide. Sulphides behaved in a similar manner, but thiophene was unaffected in contact with it. Under the conditions employed, the mercaptans were removed more readily than the sulphides and thiophene was not affected.

Experiments were carried out with the naphtha containing thiophene and the propyl sulphide-naphtha solution, in which hydrogen was added to the vapour. The results indicate that the addition of hydrogen effects the removal of thiophene in contact with the catalyst at the steady state. The addition of hydrogen also effected the removal of a larger percentage of propyl sulphide sulphur than is effected when no hydrogen is added. When the catalyst, which had been used for some time, was heated in the presence of hydrogen a copious evolution of hydrogen sulphide was produced.

Adsorption in the liquid phase showed that the mercaptans were taken up to about the same extent, and that propyl sulphide and thiophene are adsorbed to approximately the same degree. The amount of mercaptan sulphur adsorbed is, however, approximately twice that adsorbed in the case of propyl sulphide or thiophene.

These results readily account for the difficulties in sulphur removal observed with the naphthas presented in Part I. The mercaptans most readily eliminate sulphur as hydrogen sulphide, while the removal of thiophene presents a much more difficult problem by this method. Since

it is known that the thermal decomposition of mercaptans and sulphides result in the formation of thiophene, it appears in general that the sulphur content of cracked naphthas will be more difficult to reduce by the present method than will that of straight-run naphthas.

W. D. S.

278. Origin and Refining of Sulphur Oils. W. Friedmann. *Oil and Gas J.*, 1930, 24 (31), 32, 106, 107.

An extensive investigation has been carried out by the author in order to investigate the manner in which sulphur occurs in crude petroleum, shale oil, tar, asphalt, etc., from a consideration of the reactions taking place between sulphur and the paraffin, naphthene, olefine, terpene, diolefine and aromatic hydrocarbons present in such products. Great difficulty was experienced owing to the difficulty of obtaining pure materials, the lack of previous information on this problem and the intolerable odour of the lower sulphur compounds formed during the reactions.

Paraffin hydrocarbons react vigorously with sulphur under pressure at temperatures above their boiling points, splitting off hydogen sulphide and low boiling mercaptans. No olefines were found to be formed, contrary to previous belief. The residue is a coke-like mass from which only a portion of the sulphur can be extracted, pointing to the formation of a solid carbon-sulphide. No asphaltic products are formed, as is the case with olefines and naphthenes, and no liquid mercaptans could be isolated, the higher paraffins being chiefly formed (by polymerisation).

Olefine hydrocarbons when heated with sulphur react readily and polymerise, changing into ring compounds and eventually yielding asphaltic substances containing 16–17% sulphur.

Naphthene hydrocarbons react with sulphur, which can both enter the cyclohexane ring as well as the side chains. The first reaction is to dehydrogenate the ring, the unsaturated compounds formed then polymerising to form asphalts, as in the case of the normal olefine hydrocarbons. Thiophenes and diolefines also polymerise in the presence of sulphur very readily to give asphalts.

It is believed (Sakhenov. *Petroleum*, 1925, **1**, 441; 1927, **1**, 618) that if paraffins and naphthenes predominate in an oil, the asphaltenes form coarse suspensions, whereas if aromatics or petroleum resins are the chief constituents, the asphaltenes form finely dispersed colloidal solutions.

Heavy crude oils, having high resin contents, therefore have high asphaltene contents, and light crudes low asphaltene contents. It is assumed that the neutral petroleum resins originally present which are sulphur-free change under the action of oxygen or sulphur to form asphaltenes, and to other colloidal modifications, carbenes and carboids. The asphaltenes absorb large proportions of petroleum resins, which in turn will change to further asphaltenes, etc. Asphalts can therefore be described as colloidal solutions of hydrocarbons of high sulphur content in hydrocarbons of lower sulphur content (unchanged petroleum resins). In a paraffinous crude, asphaltenes will not be produced unless naphthenes are present at the same time.

C. L. G.

279. Organic Sulphur Compounds. XVII. Sulphur Analogues of Hexaphenylethane and Triphenylmethyl. A. Schönberg and T. Stolpp. *Annalen*, 1930, **483**, 90–107.

Treatment of phenylthioldiphenylmethyl chloride with mercury in the presence of benzene out of contact with air and light gives α– tetraphenylethylene and diphenylthioldiphenylmethane. Reaction is presumed to occur through the intermediate free radical phenylthioldiphenylmethyl

$$4\,CPh_2SPh \longrightarrow CPh_2{=}CPh_2 + 2\,CPh_2(SPh)_2.$$

Similar treatment of o–nitrophenylthiodiphenylmethyl chloride yields benzophenone, di–o–nitrophenylthioldiphenylmethane, and di–o–nitrophenyl disulphide; much resinous material is produced. Diazofluorene and sulphur phenyl chloride in ether give 9–chloro–9–phenylthiolfluorene, converted as above into αβ–diphenylthiol–αβ–di (diphenylene) ethane. This has a normal molecular weight in chloroform, and, when heated in the presence or absence of a high-boiling solvent, decomposes into diphenyl disulphide and di (diphenylene) ethylene. The solid is stable to light, but exposure of a benzene solution to ultra-violet light results in the formation of di (diphenylene) ethylene. The compound 9 chloro–9–o–nitrophenylthiolfluorene is converted by mercury in benzene into a αβ–di–o–nitrophenylthiol–αβ–di (diphenylene) ethane of m.p. 165–167°, which, when heated in boiling xylene or alone at 170–190°, decomposes into fluorenane and di–o–nitrophenyl disulphide. The above results show that intermediate radical formation occurs when the arylthioldiphenylmethyl chlorides are treated with mercury; some form of stabilisation of the radical then takes place.

The compound 9 : 9–dichlorofluorene and thiophenol react in boiling benzene in a current of carbon dioxide, yielding 9 : 9 diphenylthiolfluorene, which melts at 115°, and decomposes quantitatively at 240°, in a vacuum into diphenyldisulphide and di (diphenylene) ethylene. 9 : 9 di–o–nitrophenylthiolfluorene of m.p. 159–160° (from 9 chloro–9–o–nitrophenylthiolfluorene and o–nitrophenylmercaptan in benzene), decomposes explosively at 200° in a vacuum forming fluorenane, di–o–nitrophenyl disulphide, and much carbonaceous material. Similar decomposition of di–o–nitrophenylthialdiphenylmethane produces benzophenone, a small amount of di–o–nitrophenyl disulphide and a considerable quantity of carbonaceous material. W. S. C.

280. Origin and Decomposition of Organic Sulphur Compounds Under Gas Making Conditions, With Particular Reference to the Role of the Carbon-Sulphur Complex. J. C. Holtz. *Fuel*, 1931, **10**, 16–30.

The paper opens with an historical survey of the reactions between carbon and sulphur, particularly the solid residues. These may contain as much as 37·6% of sulphur.

In the preliminary experiments, gas oil containing 3·61% of sulphur was cracked in a laboratory apparatus which is described and illustrated. The gas produced was collected and examined for CS_2 by Huff's method (J. Amer. Chem. Soc. 1926, **48**, 81). It was found that at temperatures about 1200° F. no CS_2 was detected, but above this gradually increasing amounts were formed.

In further experiments, a sulphur free medicinal paraffin was cracked at 1500–1600° F. in the presence of gas from the same oil, to which

hydrogen sulphide had been added. It was established that with very high proportions of hydrogen sulphide, carbon disulpide was produced, but that the general conditions of the test favoured the loss of sulphur to compounds other than carbon disulphide.

Nitrogen, to which known amounts of hydrogen sulphide were added, was passed over sulphur-free charcoal heated to 1500° F. At first the sulphur compound was completely absorbed on the carbon surface, but gradually hydrogen sulphide passed through, and ultimately carbon disulphide was produced.

The tendency for heated surfaces to decompose organic sulphur compounds was examined by passing the high sulphur cracked oil gas over heated pumice and carbon (separately). While the surfaces were at first effective, ultimately carbon disulphide was produced.

The conclusion is reached that the carbon disulphide in coal gas is derived from the decomposition of the solid complex of carbon and sulphur.

A quantitative examination was made of the hydrogen sulphide–carbon–carbon disulphide equilibrium, by passing gas containing hydrogen sulphide over charcoal maintained at a definite temperature. The issuing gases were tested for carbon disulphide and total organic sulphur.

The experiments and apparatus are described in detail, with typical results.

The hydrogen sulphide and carbon disulphide were first estimated in the issuing gases—then after removal of the former, the organic sulphur was determined by passing the residue with hydrogen over a heated platinum spiral and estimating the hydrogen sulphide by titration with Iodine in a Tutwiler burette. The sulphur in the charcoal residue was determined by fusion with sodium peroxide and estimating the sulphate formed. The carbon disulphide was estimated by Huff's method (see above).

It is shown that organic sulphur compounds other than carbon disulphide are formed by the decomposition of the carbon–sulphur complex, but they were not identified.

The investigation was concluded with a study of the effect of varying the conditions upon the formation of organic sulphur.

An increase in temperature or hydrogen sulphide concentration, caused an increase in the concentration of both total organic sulphur and carbon disulphide. Water vapour reduced the concentration of carbon disulphide but did not appreciably affect the total organic sulphur. Carbon monoxide and oxygen caused an increase principally in the compounds other than carbon disulphide. Hydrogen caused a decrease in concentration of the organic sulphur. C. C.

281. Action of Aromatic Alcohols on Aromatic Compounds in the Presence of Aluminium Chloride. V., Benzylation of ortho-Cresol. R. C. Huston, H. A. Swartout and G. K. Wardwell. *J. Amer. Chem. Soc.*, 1930, **52**, 4484–4489.

An investigation on the condensation of benzyl alcohol and *o*-cresol in the presence of aluminium chloride has been carried out. The main product of the reaction is 2-methyl-4- benzylphenol which crystallises as

hard flattened needles melting at 49·5°–50·5° C. The product was identified by the following steps. The compound was heated under pressure to give the amine which on diazotisation and reduction gave a hydrocarbon which was oxidised to m-benzyl-benzoic acid. This proved that the benzyl group entered either in the ortho or para positions to the phenolic hydroxyl. Final proof of its constitution was established by the preparation of the isomeric 2-methyl-6-benzyl-phenol, m.p. 49·5°–50·5°, which has somewhat different physical properties. This isomer is also formed in very small amounts during the benzylation, together with some 2-methyl-4, 6-dibenzylphenol.

The monobromo derivatives of the two monobenzyl cresols were prepared by bromination in chloroform solution. In the case of 2-methyl-4-benzylphenol, it was assumed that the bromine entered the phenolic ring in the only available ortho or para positions. With the same assumption 2-methyl-6-benzylphenol gives 4-bromo-2-methyl-6-benzylphenol and this was proved by the formation of the same product by benzylating 2-methyl-4-bromophenol by the Claisen method.

The two monobenzyl derivatives were further characterised by the preparation of their benzoyl derivatives. W. D. S.

282. Decomposition of Ethylene Glycol in Presence of Catalysts. I. Vanadium Pentoxide as Catalyst. N. L. Drake and T. B. Smith. *J. Amer. Chem. Soc.*, 1930, **52**, 4558–4566.

This work was undertaken in order to test the possibility of the generation of an ethylene oxide linkage by the direct addition of oxygen to the double bond. It is shown that there is nothing thermodynamically unsound in an attempt to obtain ethylene glycol by such a method. In order to test catalysts for this synthesis their effect on the decomposition of ethylene glycol under various conditions has been examined. Gas analysis of the products of decomposition enables the course of the reaction to be followed easily. The apparatus used and the method of operation are described fully. The extended use of vanadium pentoxide as an oxidation catalyst led the authors to choose it for use in their preliminary studies.

Graphs are shown of the mol. percentage of glycol decomposed and mol. percentage of decomposition products plotted against temperature as abscissæ. From the slope of curves it is evident that there is little to be gained by operating at temperatures above 400°. It is also significant that up to a temperature of about 317° more ethylene than any other product (58% of the decomposed glycol) was produced. The curves for acetalydehyde and ethylene reach a maximum and a minimum, respectively, in the neighbourhood of 357°. Moreover, ethylene formation was more favoured by the presence of fresh vanadium pentoxide than by used catalyst, but either contact mass produced acetaldehyde in about equivalent amounts.

Among the other products small amounts of carbon dioxide and carbon monoxide, acetic acid and methane were detected. A mechanism of the reaction to account for the presence of all the decomposition products is suggested and it is probable that the decomposition passes through the ethylene oxide stage. W. D. S.

283. Formation of Di-isobutylene from Acetone at High Pressures. A. D. Petrov. and F. Z. Ivanov. *J. Russ. Phys. Chem. Soc.*, 1930, 12, 1489–1492.

By heating acetone in the presence of zinc chloride under pressure at 300° C., a mixture of unsaturateds is obtained of which a large proportion is di-isobutylene (contained in the fraction 100°–110° C., d_{18}^{18} 0·7198 n_{γ}^{18} 1·416). Hydrogenation of this compound gave a good yield of $\beta\beta$-trimethylpentane (b.p. 100° C., d_{20}^{20} 0·6959 η_{γ}^{19} 1·397) which is useful as a standard of comparison for the anti-knock properties of motor fuels. The bearing of the results on the mechanism of formation of petroleum is discussed. W. H. T.

284.—Catalytic Action at High Temperatures Under High Pressures. II. Catalytic Hydrogenation of Aromatic Nitro-Compounds. G. Komatsu and R. Amatatsu, *Mem. Coll. Sci. Kyoto*, 1930, A13, 329–336.

Aniline is obtained by the reduction of nitro benzene with hydrogen in the presence of reduced nickel at 100° C./50 atm. At 200° C./100 atm., however, a mixture of *cyclo*-hexylamine (78%), *dicyclo*-hexylamine (7%), and aniline (5%) is obtained. Similar results may be obtained from aniline at 180°/100 atm. p-Nitrophenol is reduced in the same way (100°/50 or 100 atm.) to p-aminophenol, and at 160°/30 or 100 atm. The main product is 4-aminocyclohexanol, together with traces of p-aminophenol, *cyclo*-hexane, hexanol and ammonia. α-Naphthylamine and ac-tetrahydro-α-naphthylamine are obtained by reduction of 1-nitronaphthalene at 50°/30 or 100 atm. and 150°/30 or 100 atm. respectively. W. H. T.

285. Vapour-Adsorption Capacity of Silica Gels. H. N. Holmes and A. L. Elder. *J. Physical Chem.*, 1931, 35, 82.

It had been noted that there is a tendency for the more porous silica gels to decrease in adsorption capacity on standing for a few years, and hence the benzene-adsorption capacities of some gels prepared several years ago was redetermined. Eleven such gels were examined, and all showed a decrease in benzene-adsorption capacity. Four of the five examined were heated above 900° C. without any serious decrease occurring in their benzene-adsorption capacity. By increasing the temperature of acid treatment of a gel from 30° C. to 100° C. the porosity of the gel is increased. A gel capable of absorbing 156% of its own weight of benzene from an air stream saturated with benzene at 30° C. was obtained.

The most porous gels, which still had a good firm structure, were obtained from gels dried to 50–60% moisture content before acid treatment. Useful gels may be made by first drying to a water content as low as 30%.

The Holmes' chalky gels have large capacities for adsorbing benzene under high partial pressures, but are not efficient under low partial pressures. The Holmes' vitreous gels are practically equal to the hard

glassy type at low partial pressures, yet have double the total capacity of the later at high partial pressures.

Increase in efficiency of vapour adsorption, by utilising chalky gels to remove most of the vapour, followed by use of vitreous or glassy gells to remove the last traces is suggested. H. E. T.

286. Some Unusual Properties of Colloidal Dispersions. R. V. Williamson. *J. Physical Chem.*, 1931, **35**, 354.

Experiments are described which show the marked changes in the plastic and elastic properties of dispersions, which may be produced simply by changes in temperature, or in the magnitude and the rate of application of stress. The experiments may be useful to manufacturers of colloidal materials, since they illustrate some of the unexpected results that may occur from simple changes in the physical environment of the materials. They should also be useful to those interested in the study of structure in colloidal dispersions, since they furnish a number of new facts which must be explained by a comprehensive theory of colloidal structure. H. E. T

287. Method of Measuring Average Particle Size of Emulsions. W. P. Davey. *J. Physical Chem.*, 1931, **35**, 115.

A method is described for measuring the average particle size of oil-water emulsions.

An enamelled iron tray about 8 in. wide, 30 in. long, and ¼ in. deep is filled with water. A piece of paraffined aluminium foil is floated on the surface at one end of the tray and attached to a balance such as was used by Langmuir, or to a torsion wire such as is used in the Du Nouy apparatus. The spaces between the walls of the tray and the ends of the aluminium float are sealed by constant pressure air jets. The remainder of the water surface is then swept free of monomolecular layers of grease, oil, etc., by means of glass sweepers, and a drop of the emulsion is spread on this clean surface from a micrometer pipette. The area of the film is determined exactly as in Langmuir's experiments with oil films.

The calculation of particle size requires a knowledge of the total volume of the droplets of the disperse phase as they exist in the emulsion. The volume may be obtained by curdling a known volume of emulsion with a known volume of a solution of a suitable electrolyte, removing any included water from the curd and adding it to the rest of the water phase. The total volume of the water phase is then measured and the volume of the disperse phase is found by difference. This gives the concentration C of the emulsion. If V is the volume of the drop of emulsion which was spread on the water, then C V is the volume of disperse phase in that drop. The mean diameter of the particles of disperse phase is therefore—

$$d = \frac{CV}{A}$$

where A is the area which was covered.

The various limitations to which this method is subject are then discussed in detail. H. E. T.

288. Preparation of Organic Compounds from Hydrocarbons having One Double Bond in the Molecule. H. D. Elkington and N. V. de Bataafsche Petr. Mij. E.P. 339,592, 6.8.29.

Cyclic hydrocarbons, having one double bond in a non-aromatic nucleus, are absorbed in H_2SO_4. Catalysts for the process are described in Specification Nos. 323,748, 336,603, 336,604 and 336,633. The absorption products are subjected to treament such as hydrolysis and distillation for the production of compounds such as alcohols, esters or ethers. An example is given of the absorption of cyclohexane at very low temperature in 80% H_2SO_4 containing a small amount of ferricyanic acid to give cyclohexanol. W. D. S.

See also Abstract No. 196.

Analysis and Testing.

289. Errors in Analytical Bromination. New Procedure for Determination of Organic Unsaturation. H. M. Buckwalter and E. C. Wagner. *J. Amer. Chem. Soc.*, 1930, **52**, 5241–5254.

Halogenation methods for the determination of organic unsaturation are discussed. Both addition and substitution of halogens are influenced by a variety of factors. They include not only time, temperature, and quantity and concentration of halogen, but also identity of solvent condition in which halogen is present, proximity to unsaturated linkage of strongly polar radicals or other structural influences, and presences of substances which may operate catalytically. Several sources of error, such as formation of halogen acids by contact with water, replacement of chlorine or bromine in contact with alkali iodine, reversible cleavage of hydrogen chloride after addition of iodine chloride to certain α,β– unsaturated acids, partial ester hydrolysis, presence of an acidic function in the substance analysed, rupture of a chain or bridge followed by additive halogenation, cleavage of hydrogen halide, formation of perhalides, are discussed.

The method adopted for the determination of organic unsaturation is a modification of the method of McIlhiney in which the compound is brominated in carbon tetrachloride and the bromine of substitution estimated by shaking with alkali iodide solution. In the present method, contact with water is avoided, the excess bromine and hydrogen bromide being removed by aeration with nitrogen and absorbed in potassium iodide solution. Brominations were conducted at 75°, at 20–25° and at 0°C. About forty organic substances of various kinds, both unsaturated and saturated, were examined. Comparative analyses were in all cases made by the method of McIlhiney. Tables are given showing total bromine consumed, in addition and substitution.

In all but a few cases the aeration method, under suitable temperature conditions, yielded better results than did McIlhiney's method. Distinct temperature effects were noted for hydrocarbons such as cyclohexene, phenanthrene, indene and especially retene. Styrene and stilbene gave values for bromine addition little affected by temperature. It was noted that by proper temperature control of the brominations negative results could be completely eliminated.

The aeration procedure has yielded an explanation for the low or negative bromine values obtained for certain substances, since by exclusion of other sources of error it has brought into prominence the spontaneous cleavage of hydrogen bromide (other than that attributable to substitution) from such substances when exhaustively brominated at too high temperatures. This conclusion was verified by experimental elimination of hydrogen bromide from several substances brominated at low temperature and then warmed to 75°C.

Results obtained with several technical hydrocarbon mixtures from petroleum–heavy lubricating oil, transformer oil and cracked gasoline–are recorded. The unsaturation of several compounds of acidic character has also been determined and since the errors from such compounds are usually only operative in water the advantage of the aeration method in this case is obvious.

The practical application of the findings by the aeration procedure requires the use of bromination temperatures low enough to prevent excessive consumption of bromine by reactions other than addition. It was found that some substances brominate normally at 75°, others at room temperature, while others yield entirely meaningless results unless brominated near 0°, which for some substances is perhaps still too high a temperature. The new procedure permits the necessary temperature regulation and avoids complications due to contact with water, iodide and iodate with the brominated sample. W. D. S.

290. Meaning of Gasoline Distillation Curve. G. Edgar, J. B. Hill and S. A. Boyd. *Oil and Gas J.*, 1930, **29** (26), 144, 256.

Motor fuel volatility is related to engine performance chiefly in the following ways:—(1) Ease of starting a cold engine; (2) tendency to interrupt operation because of vapour lock; (3) ease of acceleration, particularly during the warming up period; (4) relative ease of effecting a dry mixture; (5) tendency to crank case dilution.

It has been shown that if the A.S.T.M. distillation curve of a fuel is known, its characteristics with regard to all five of these factors may be at least approximately calculated.

A study of the fundamental principles involved leads to several practical principles which may serve as a guide to refiners in indicating the volatility characteristics which gasoline should possess to give satisfactory engine performance under various conditions, and to the automobile engineer in indicating methods by which the fuel feed systems of automobiles may be improved. W. L.

291. Modify Doctor Test Procedure for Use on Natural Gasoline. *Oil and Gas J.*, 1931, **29** (33), 105, 107.

Since the present method is unreliable, the Natural Gasoline Association of America has adopted a revised method for doctor test as part of its standardisation programme. The reagents employed are as follows:—(1) Sodium plumbite solution containing 125 g. of NaOH and 25 g. of PbO per litre; (2) pure dry flowers of sulphur ground and screened to 100–200 mesh. The reagents are to be chemically pure and must be stored in clean, tightly closed receptacles.

A special method for the preparation of the sodium plumbite solution is recommended. The test is carried out in standard cylindrical 4 oz.

sample bottles, having an internal diameter of approximately 33mm., fitted with clean cork stoppers.

10 ml. of the sample to be tested is added to 5 ml. of plumbite solution in the standard bottle, which is tightly stoppered and shaken vigorously for 15sec. Just sufficient sulphur is added (25–30 mg.) to cover the interface between the sample and solution. The bottle is then stoppered and shaken vigorously for 15 sec., after which the contents are allowed to settle.

The sample is reported as "sweet" if there is no discoloration of the sulphur film, and "sour" if the film immediately becomes discoloured or flecked with brown or black. Indications of (1) H_2S, (2) elementary sulphur and mercaptans, and (3) mercaptans and absence of H_2S and elementary sulphur, are obtained on adding the sample to plumbite solution and observing respectively: (1) Immediate black precipitate, (2) opalescence and darkening of colour of sample after shaking, (3) clear yellow solution. R. A. E.

292. Determination of Lead Tetraethyl in Gasoline. L. J. Catlin and J. E. Starrett. *Chemist-Analyst*, 1930, **19**, (5), 5–6.

A 10% solution of bromine in carbon tetrachloride is added slowly, with stirring, to 100 c.c. of the sample to the extent of 10 c.c. for straight run spirit and 30 c.c. for cracked spirit. After a few seconds, 5 c.c. are added in excess, the lead bromide allowed to settle, filtered off and washed by decantation with 25 c.c. of CS_2. The precipitate is then washed back into the flask with, and dissolved in, 30 c.c. of hot 15% nitric acid, 5 c.c. of concentrated H_2SO_4 are added, and the mixture heated until white fumes appear. The lead sulphate is filtered off, washed, dissolved in hot ammonium acetate solution and titrated while hot with ammonium molybdate solution (8·6 gm./litre, 1 c.c. = 0·01 gm. of lead). A yellow colour with a fresh 0·5 % solution of tannin, used as outside indicator, gives the end-point. W. S. C.

293. Measuring Unconsumed Gases in Exhaust. *Aviation*, 1931, **30**, 57.

An instrument known as the "Moto-Vita" for indicating the amount of unconsumed gases in petrol-engine exhaust gases has been developed in the laboratory of Dr. M. R. Hutchinson.

The instrument detects any combustible gas (CO, H, or gasoline vapour) which may be present in the exhaust. A table is given showing the correspondence between CO content, air-fuel ratio and completeness of combustion, the figures varying from 11·8% CO with 9·6 air-fuel ratio, and 52% combustion to 4·2% CO with 13·2 air-fuel ratio and 82% combustion.

The "Moto-Vita" consists of a Wheatstone bridge, battery and milliammeter. The alternate arms of the bridge consist of nickel and platinum wires arranged to have equal resistances in each of the four arms at equal temperatures.

Exhaust gases are passed over these wires. Catalytic oxidation of carbon-monoxide occurs on the surface of the two platinum arms of the bridge, thus upsetting the balance and causing a deflection of the milliammeter.

The dial is calibrated to show weak, correct and rich mixtures The instrument has been tested out on aeroplanes during the past year and appreciable fuel savings have been achieved. E. B. E.

294. Determination of Corrections for Redwood Viscometers. G. Barr. *J. Sci. Instr.*, 1930, 7, 359.

The equations $\nu = AT - B/T$ and $\nu = A^1T^1 - B^1/T^1$ are assumed to hold for the standard Redwood viscometer at the N.P.L. and for an instrument under test respectively, where ν is the kinematic viscosity and T and T^1 are the times of flow of 50 ml of oil. Parallel flow tests are made with two oils at room temperature, and the results are used in a simple graphical method to estimate the corrections $T^1 - T$, which should be applied at various values of T. A. R. S.

295. New Pipette Viscometer. J. Romney. *J.S.C.I.*, 1930, 49, 986-988.

The apparatus consists essentially of a tube drawn off at its lower end and fitted with a stopcock. Inside this tube is fixed a glass overflow gallery and also a tube dipping below this level, and connected through the outer tube to a pipette with two level marks. Oil is drawn through the stopcock into the main tube up to the overflow, thus ensuring a constant head for the pipette, which is now filled and the time of flow noted between marks.

In calibration of the instrument a series of oils of known Redwood viscosities is run through the pipette and a factor calculated.

It is admitted that the conversion factor is not a constant for all types of oil.

Adequate temperature control of a number of these pipettes can be obtained in one thermostat, and cleaning by use of an easily volatilised solvent is claimed to obviate removal from the thermostat. A. R. S.

296. Specific Heat Charts for Gases. A. C. Halferdahl. *Chem. and Met. Eng.*, 1930, 37, 686-687.

Calculations of mean specific heats of gases are shortened by means of graphs appended to the paper. The data given are based on Eastmans specific heats and are summarized as follows—

	Temp. Range degrees Kelvin.
Hydrogen.	
$Cp = 6{\cdot}85 + {\cdot}00028T + {\cdot}000000327T^2$	300-2500
Nitrogen, Oxygen and Carbonmonoxide—	
$Cp = 6{\cdot}76 + {\cdot}000606T + {\cdot}00000013T^2$	300-2500
Carbondioxide and Sulphur Dioxide—	
$Cp + 7{\cdot}70 + {\cdot}0053T + {\cdot}00000083T^2$	300-2500
Water Vapour—	
$Cp = 8{\cdot}22 + {\cdot}00015T + {\cdot}000000134T^2$	300-2500
Hydrogen Sulphide—	
$Cp = 6{\cdot}70 + {\cdot}0036T$	300- 600
Methane—	
$Cp = 5{\cdot}90 + {\cdot}0096T$	300- 800
Ammonia—	
$Cp = 6{\cdot}70 + {\cdot}0063T$	150- 400
Chlorine, Sulphur vapour (S_2)	
$Cp = 8{\cdot}58 + {\cdot}0003T$	300-2500

A. R. S.

297. Oil Scrubbing Apparatus. *Report of Fuel Research Board*, March 31, 1930, 79.

The oil scrubbing of gases on a small scale has hitherto been carried out in large glass tubes filled with Lessing rings; this method has necessitated the presence of an observer for removing the used oil and replenishing the reservoir at the top of the towers. An automatic

apparatus has now been designed, consisting of three metal towers in series, in which the oil is circulated in counter flow to the gas by means of small rotary pumps driven off one shaft and collected in a saturated condition in a reservoir. The apparatus does not require attention and is much clearer in operation. A fourth column for stripping the oil is to be added. H. E. T.

298. Estimation of Wax in Pitch. W. Littlejohn and W. H. Thomas. *J. Inst. Petr. Techn.*, 1930, **16**, 814–824.

A brief summary, together with comments, of some of the better known methods of estimating the wax content of petroleum residues is given. The authors have selected the cracking distillation method followed by precipitation of the wax by means of acetone, as being the most convenient for normal laboratory procedure. Experimental data are given dealing with the effects of the following variables on the final result—temperature of precipitation, extent of dilution of oil wax mixture with acetone and effect of distillation conditions. In view of the results obtained and from the conclusions arived at, a method of estimating the wax content of petroleum residues has been devised which in practice gives results with a concordancy of $\pm 0{\cdot}2\%$. The following is a *résumé* of the method—10 g. of the sample are distilled at a standard rate of rise in temperature from a 70 c.c. glass retort. The distillate obtained is weighed and dissolved in acetone to the extent of 35 c.c. per g. of distillate. The mixture is then cooled to 0° F. and the precipitated wax removed by filtratioe. The wax is removed from the filter by means of hot petroleum ether and after evaporation of the latter dissolved in acetone to the extent of 70 c.c. per g. of wax. The resulting mixture is cooled to 32° F. and the precipitated wax finally removed by filtration and weighed. The results are expressed as "percentages of wax determined at 32° F. (and/or 0° F.) precipitated from acetone," the m.p. of the final wax being included in the result.
W. H. T.

See also Abstracts Nos. 198, 211.

Engines.

299. Ruston and Hornsby High Speed Diesel Engine. *Mar. Eng.*, 1931, **54**, 12-13.

This new engine (the V.Q. type) when direct-coupled to a dynamo is suitable as a ship's generator engine, or if fitted with a pulley, for industrial purposes ashore. It operates on the four-stroke cycle with airless injection and is cold starting, automatic air-starting valves being fitted to the cylinders. It is obtainable in two, three, four and six cylinders. Each cylinder (8 in. stroke, 5¼ in. bore) yields 16 B.H.P. at 900 r.p.m. The familiar Ruston and Hornsby combustion chamber is used, the two valves being disposed horizontally with the atomiser in the centre of the head and spraying vertically downwards through the narrow combustion chamber. The valves are accessible, and danger of burning or over-heating of the piston is eliminated. The close grained cast-iron heads are separate from the liners. The sump serves to retain the lubri-

cating oil, the well at the forward end containing an oil strainer and gear wheel type forced lubrication pump, which feeds the principal bearings, a pressure relief valve serving as a means of regulating the oil pressure. The small end bearing (and the fully floating gudgeon pin) is pressure lubricated through the light "H" section connecting rod.

The fuel pump is operated by a quick acting cam through a roller working direct upon the fuel pump plunger. The centrifugal governor, gear-driven from the crankshaft, regulates the quantity of fuel according to the load requirements

Whilst the two-cylinder model can be started by a starting handle, the larger engines are fitted with air starting. The guaranteed fuel consumption is very low and operation is satisfactory on paraffin, kerosine distillate, gas oil and almost any diesel fuel oil. C. I. K.

300. Lister Airless-Injection Engine. *Engineering*, 1930, **130**, 802.

One of the principal objections to the pre-chamber design of airless-injection engine, as usually constructed, is the necessity for some auxiliary starting device to supply heat during the first few revolutions.

This has been overcome in the new Lister design. The main pre-chamber in this engine is connected by a small orifice to a smaller chamber. For starting, the latter is isolated from the pre-chamber by means of a hand-operated valve. The decrease in the total clearance volume so effected raises the compression ratio from the normal running value of 15 : 1 to the special starting ratio of 19 : 1. As soon as normal speed is attained the valve is lifted by means of a hand-wheel. It is claimed that this engine will operate on any of the usual fuels without alteration, consumption not exceeding 0·45 pt./b.h.p./hr. and exhaust clear at all loads apart from the moment of starting. G. B. M.

301. Hesselman Oil Engine for Motor Vehicles. A. Heller. *Z. Ver. deut. Ingenieure*, 1930, **74**, 970–972. *Mech. Eng.*, 1930, **52**, 1082–1083.

This is a high speed oil engine with compression ratio of only 5.5 : 1. A rotating motion of the air is obtained about the axis of the cylinder by screening off on one side the intake valve, and towards the end of the compression stroke finely atomized fuel is slowly injected into this rotating air stream, in such a manner that air flowing past the spark plug is sufficiently saturated with fuel to be easily ignited.

The interesting feature of this engine is the design of the piston, which is provided in its upper part with a ring which at the end of the compression stroke practically closes in the combustion space on the sides.

This ring has two slots—one for the spark plug and the other for the fuel nozzle situated opposite the spark plug.

The spark plug in its slot is protected against direct access of the fuel to the terminals. Pronounced turbulence is produced when the air passes the spark plug.

It is claimed that this engine will start from cold on gas oil of about 0·86 sp. gr. W. A. W.

302. Horizontal Diesel Engines. P. Kromgar. *Z. Ver. deut. Ingenieure*, 1930, 74, 1441-1442. *Mech. Eng.*, 1931, 53, 57–59.

Engines of this type have been installed in the power plant of a pumping station in Argentina, each unit consisting of a horizontal four-stroke cycle Diesel engine with airless injection and two double-acting cylinders arranged in tandem. These Diesel engines have a bore of 850 mm., a stroke of 1250 mm. and run at 125 r.p.m. Only one piston-rod is provided, to which both pistons are connected.

Starting is easily effected by admitting compressed air into the second and fourth combustion chambers, and as soon as the engine turns over fuel is admitted into the first and third combustion chambers when, after two strokes, the air is cut off and fuel injected into the other two combustion chambers. The normal operating pressure at full load is 37 atm., but the maximum ignition pressure at the beginning of fuel admission does not reach this figure.

Data is given regarding fuel consumption and cooling, also the efficiency of the current generator. W. A. W.

303. Fuel Injection of Diesel Engines. J. A. Spanogle and H. H. Foster. *Aircraft Eng.*, 1931, 3, 19-21.

This paper deals with the performance of a high speed compression ignition engine using multiple-orifice fuel injection nozzles.

The chief difficulty with this type of engine is the bringing of all the fuel into contact with sufficient air for its combustion.

An attempt was therefore made to obtain the requisite mixture by injecting the fuel through a combination of round orifices in a nozzle. Two methods were used:—1. Engine performance tests on a N.A.C.A. Universal Test Engine with a special head were carried out, systematically varying the number, direction and size of the orifices. 2. Mathematically proportioning the discharge of each orifice to the volume of air to be served by the spray from this orifice. (Photographs of the spray were taken with the N.A.C.A. spray photography equipment.) Mechanical details of the engine arrangements are given with illustrations. Nozzles with 4, 5, 7, 8 and 9 orifices were tried.

The results are given in the form of curves and a chart listing the nozzles and dimensions of orifices and the performances obtained.

It is concluded that to obtain efficient combustion in a cylinder head with low rates of air flow during the injection of the fuel, it is necessary to proportion and direct the fuel sprays so that the fuel is distributed as uniformly as possible throughout the air.

The test engine results indicate that impingement of the fuel spray on the piston and chamber walls is not necessarily detrimental to combustion, but may be an aid to distribution. C. C.

304. Progress in the Diesel Engine for Road Transport. W. H. Goddard. *Petr. Times.*, 1930, 24, 925–938.

The progress made by the Diesel Engine for road vehicles has been considerable when it is realised that it is only two and a half years since the first Diesel Engine vehicle was put on the road.

The drawbacks of noise and exhaust fumes have been overcome to a tremendous extent, and it is claimed that carbon monoxide is present

in the exhaust fumes only in the very smallest quantities, and, at certain speeds, is entirely absent.

It is logical to assume that the life of the Diesel Engine would be longer than that of the Petrol Engine for the following reasons. 1. Speed is slower and regulated by the governor. 2. Engine works under much cooler conditions. Lubricating oil retains its lubricating qualities for much longer period. 3. Practically no dilution in the sump. 4. Power stroke of Diesel more akin to the stroke of a steam engine, resulting in less shock. 5. Radiator not subjected to such violent changes in temperature as that of a Petrol Engine.

An advantage could still be shown even if the price of Diesel oil was raised to that of petrol, because of the greater mileage obtained.

The question of weight per B.H.P. needed study, and although present day weights would be reduced considerably, it is not expected, or necessary, to get down to petrol engine standards. L. W. C.

305. Specialization Will Help to Overcome High Cost of Production and Solve Metering Problems. [Overcoming the Limitations of the Automotive Diesel Engine.] P. H. Schweitzer. *Aut. Ind.*, 1930, **63**, 820–822.

The Diesel Engine outweighs the corresponding carburettor engine by 2·5 to 1. In a well-designed engine the structural weight is probably roughly proportional to the square root of the highest cylinder pressure. 100lb. sq. in. is the present practical limit of b.m.e.p. for Diesels. This drawback is inherent in the compression-ignition principle and better performance can only be obtained by better control over injection and distribution. The present practical limit for engine speed is about 2,000 r.p.m.

Injection lag and Ignition lag are the main factors which limit this speed, but the former can be allowed for by injecting the fuel proportionately earlier.

Ignition lag is the most serious limiting factor with respect to speed and output of compression-ignition engines.

For satisfactory thermal efficiency, the injection period must not exceed 40°, the ignition lag should not be more than 20% of this.

Reduction of the ignition lag may be effected by:—1. Finer atomization. 2. External source of heat, e.g., sparking plugs or hot surfaces. 3. Higher compression pressure and temperature (c.r. of 18 : 1 or 20 : 1). 4. Injection started very early but only in small quantities.

C. C.

306. Needle Bearings Offer Advantages in High-Speed Automotive Engines. M. Pitner (From paper read to the French Society of Automotive Engineers). *Aut. Ind.*, 1930, **63**, 869–870.

The needles of the bearings differ from the usual roller bearings in that they are held endwise by shoulders and are not guided by a cage. They are made in diameters of from 0·080″ to 0·160″ and loads as high as 7800 lb./sq. in. have been carried. Loads of 3400 lb./sq. in. are regularly carried at 5000–8000 r.p.m.

When such a bearing begins to operate, the needles and raceway are in direct contact. Under the effect of friction between the needle and the raceway, the former begins to roll; oil immediately enters

between the needle and the raceway and forms a film, completely surrounding the needles.

Since the force of friction of the needles against the prism of oil imprisoned between adjacent needles is the predominating force, the needles cease to roll, and the assemblage forms a kind of intermediate sleeve, similar to a floating bush.

Under sudden increase of load the film between needle and raceway may be cut, whereupon the friction causes the former again to begin to roll. At such moments the load is carried not only by the needles along their line of contact but also by part of the lubricant between the needles.

This type of bearing is particularly useful in a high-speed engine, *e.g.*, on crank pins, where ordinary anti-friction bearings cannot be used owing to the large increase in the centrifugal force which would result therefrom. C. C.

307. Engineering Problems of Modern Motor-Cars. W. G. Wall. *Mech Eng.*, 1930, 52, 1070–1074.

Mention is made of the number of cylinders fitted to-day, even up to 16; also of the increased engine speed and of front-wheel drive.

Aluminium pistons of the split-skirt type possess the advantage that they can be fitted fairly tightly when the cylinder is cold, yet scoring will not occur when the piston expands on heating.

Description is given of various types of valves, including a hollow-stemmed airplane-engine valve, tungsten-lipped with the hollow filled with salt; also a salt-cooled motor-bus valve with a hardened tungsten plate at the bottom of the stem. This is filled with nitrate of potash.

The design of manifolds is discussed, preference being given for those with rounded corners instead of perfect right-angle corners. It is found that the latter produces undue friction and tends to hold back the charge, making it impossible to get the maximum power out of the engine.

Regarding combustion chambers, of which many kinds are described, the compact types give higher efficiency, indicated by the T-head engine practically having gone out of existence and the valve-in-the-head-engine becoming more popular.

The path of travel of gas entering the cylinder and turbulence are problems intimately associated with the shape of the combustion chamber; also, at least a part of the cool gas coming in through the intake valve should pass over the exhaust valve.

Notwithstanding the various possible shapes of combustion chambers, the efficiency depends on the location of the spark plug. The best location for the spark plug for power, in the L-head type of combustion chamber, appears to be the centre of a triangle formed by lines running through the centres of the valves and the near edge of the cylinder. W. A. W.

308. Motor Ships and Marine Oil Engines in 1930 (No. 1). *Engineer*, 1931, 151, 9–11.

Last year about half the motor vessels built throughout the world were built in British yards, and the orders placed for motor ships exceeded those for steam ships both at home and abroad. The main

feature of the year was that almost half the ships completed were oil and molasses-carrying tankers.

Particulars of the British built motor ships completed during 1930 are given, including speed and type, type of propelling machinery, and the main engine particulars. A table is given showing the number of British built motor ships completed, their total tonnage and I.H.P., for each year of the last ten years. J. M. F.

309. Car Designer Can Solve Vapour Lock Gasoline Problem. *Oil and Gas J.*, 1930, **29** (30), 181.

At a recent meeting of the Philadelphia section of the Society of Automotive Engineers, Dr. O. C. Bridgeman gave as the essential properties of a gasoline easy starting, rapid acceleration, good distribution, low dilution of crankcase oil, freedom from corrosive sulphur, high anti-knock value, absence of gum forming constituents and of tendency to vapour lock. Owing to the very wide variations in design of present-day cars, the refiner cannot meet all these requirements for every engine and has to effect a compromise. Dr. Bridgeman said that at present gasoline temperatures of 200° F. have been observed in some fuel systems and this restricts the use of natural gasoline in motor fuels, thereby causing a conflict between ease of starting and tendency to vapour lock. Car designers could assist in solving this problem by designing fuel systems in such a way that the maximum gasoline temperature in the system is never more than 30° above atmospheric temperature. Better distribution could also be obtained by improvements in manifold design and fuels of lower anti-knock value could be employed if improvements were effected in combustion chamber design. R. A. E.

310. Vaporizers for Internal Combustion Engines. G. M. Blackstone, F. Carter and E. Carter. E.P. 337,962, 31.10.29.

This invention relates to liquid fuel vaporizers for internal combustion engines, particularly for the type in which the mixture of air and fuel is drawn through a heated passage in the induction system. In such passages only the outer portions of the column of mixture are heated sufficiently to vaporise the fuel constituent, so that a large amount is carried through to the cylinder in the liquid form, where it readily deposits on any surface on which it may impinge. In order to obviate this defect, it is proposed to provide a number of strips or columns of suitable section, which are cast integral with and connect the opposite walls of the vaporising passage. These strips or columns will conduct the heat from the walls to the middle of the passage, so heating the central portion of the mixture stream. G. B. M.

311. Treating Exhaust Gases of Internal Combustion Engines to Render them Innocuous. L. Gaisman and S. H. Peachey. E.P. 338,933, 24.7.29.

This invention comprises the process for the catalytic conversion into carbon dioxide of carbon monoxide contained in the exhaust gases emitted from internal combustion engines, wherein air is introduced under pressure into the exhaust system, at, or adjacent to the engine exhaust manifold, to mix with the exhaust gases, and the mixture passed

direct to a " palladised " carrier contained in a chamber enclosed by a heat-retaining medium so that the heat of the exhaust gases is retained to initiate the said catalytic conversion, and the heat of such reaction conserved to maintain the same. The following comparative table of results, obtained from tests on a motor vehicle, shows the percentages of the various constituents of the exhaust gases before and after applying this process.

Sample A without the catalyst. Engine runing at a speed equivalent to 20 m.p.h. Sample taken after the engine has been running for some time.

Sampe B with the catalyst. Engine running as for A. Extra air admitted to exhaust manifold. Other conditions unchanged. Sample taken after running for 15 min.

Sample C, taken 18 min. after B.

Sample No.	A.	B.	C.
Carbon dioxide	5·07	8·32	12.75
Unsaturated hydrocarbons	Nil	Nil	Nil
Oxygen	5·42	8·66	4·93
Carbon monoxide	9·36	1·60	1·81
Hydrogen	8·41	1·57	1·69
Methane	9·56	1·82	1·71
Nitrogen (by diff.)	62·36	77·83	77·61
Total	100·00	100·00	100·00

In the above tests no special conditions were introduced except as were stated. Initial heating of the air as it mixes with the gas is an important feature, since admission of cold air would check the action of the catalyst By employing a pump driven by the engine, the supply of air may be regulated automatically with the speed of the engine.

H. E. T.

See also Abstract No. 293.

General.

312. Many Products from Burmah Crude Oils. G. Egloff. *Oil and Gas J.*, 1930, **29** (30), 46, 133.

Oil was produced as long ago as 1797 in Burmah, but it was not until 1889 that the first well was drilled by machinery. In 1928 the crude oil produced was about 240,000,000 imp. gal. Geological and production data for the various fields are given and a table of the total annual production of Burmah for several years during the period 1888-1927.

The crude oils of Burmah vary widely in character, but are mainly composed of paraffin and naphthene hydrocarbons. The average crude yields 28% of gasoline, 51% of kerosine fractions, 8% of wax, 3·5% of lubricating oil, 8% of residue.

The gasoline, which needs no refining, contains roughly 2·5% of benzene, 5·2% of toluene, 6% of xylene.

An ultimate analysis of the gasoline showed 85·8% of carbon, and 14·2% of hydrogen.

The ultimate analysis of the kerosine fraction is given as 86·5% of carbon, 13·5% of hydrogen. Two-thirds of this kerosine fraction is filtered through Bauxite in order to produce a water-white product, and the remainder is sold untreated as a second quality product.

The wax distillate is treated with soda, refrigerated and filtered, and the crude scale wax is sweated and filtered over Bauxite. Grades of wax with melting points ranging from 120° to 145° F. are marketed. By fractional crystallization a number of paraffin hydrocarbons, many of which correspond to those obtained from other oils and identified by earlier investigators, have been prepared.

The lubricating oil fraction is treated with acid, followed by a vacuum distillation, yielding oils of the required specifications. The lubricating oils possess relatively high specific gravities.

The crude oils from Yethaya and Palanyon are practically free from asphaltum and yield petrolatum as a residue. The production from these areas is relatively small.

Cracking experiments conducted on the residue from atmospheric distillation of a mixture of Yenangyaung and Singu crude oils, using the non-residuum method, yielded about 62·3% of pressure distillate (from which 47% of gasoline having a F.B.P. of 428° F. and 15% of furnace oil, were obtained), and 37·7% of gas, coke and loss. Details of the operating conditions are given, and it is stated that the coke yield is higher than is usually obtained when crackng other oils. The pressure distillate is treated with sulphuric acid, water and soda, and then steam distilled to produce a finished gasoline having the following properties:—A.P.I. gravity, 57·2°; I.B.P., 118° F.; 20% distilling to 211° F.; E.P., 434° F.

There are nine refineries in British India, the largest of which is at Syriam, and has a daily capacity of 16,500 brls. Cracking plants are in operation at Syriam, and others are under construction at Digboi, Assam. R. A. E.

313. Improved and Speedier Tankers Used. *Oil and Gas J.*, 1930, 29 (30), 154, 216, 218.

The expansion of the world's petroleum production and marketing organisation has necessitated a great increase in the tanker fleet, so that at present tanker construction represents 40% of the world's total ship construcion.

In 1922 ocean tankage represented 7,000,000 tons dead-weight; while at present the estimate dead-weight is 13,000,000 tons with 2,000,000 tons under construction.

The S.O.C., New Jersey, operates the largest tanker fleet in the world, having a dead-weight of 1,350,000 tons with 240,000 tons under construction This represents a capacity of 10,500,000 barrels. Royal Dutch Shell and subsidiaries' fleet represent some 950,000 tons dead-weight and Anglo-Persian some 850,000 tons.

The control of the tanker fleets is passing more and more into the hands of the large operating companies, leaving private operators in an unfavourable position for competing in business. The great increase in tanker construction is gradually approaching saturation point. As a result of this, combined with world conservation of petroleum, the freight rates have fallen very heavily, the basic rate for the Gulf-U.K. run being now 10s. 6d. per ton as compared with 22s. 7d. per ton eight years ago.

Improvements to tanker design have been made to increase speed and capacity, the former by the use of modern Diesels or high pressure

steam turbines, so that modern tankers are capable of making 11/12 knots as against the 9/10 knots of a few years ago.

The largest tanker in the world is the C.O. Stillman. It has a deadweight of 22,700 tons, lengths of 565ft. and beam of 15ft., and draws 31ft. water when fully loaded.

One of the biggest problems in tanker service is corrosion, owing to the increased amount of light products, including benzol, now handled by tankers designed to take heavier products. This is to some extent being counteracted by the use of special steels in construction.

Recent regulations bring the American tanker fleet into line with other shipping interests in the matter of load line. This will reduce the permissible amount to be carried by American tankers, no load line being previously fixed for American tankers, although laid down for other countries' ships. C. L. G.

314. Causes of Failures of Cable Couplings. S. Gasarian and V. Krasuski. *Azerbaijan Oil Ind.*, 1930, No. 10, 125–129.

Investigation of causes of failures of cable couplings shows that insulating compound of high m.p. (180° C.) is unsatisfactory. Use of such compounds leads to the formation of cavities and partial decomposition of paper and rubber used for separating the strands. It is difficult to prevent resins separating from the compound under the influence of electric field. Comparatively heavy weight of the couplings leads to mechanical deformation of electrical cables, with consequent failures. It is proposed to alter the construction of the couplings to allow of the use of semi-fluid compounds similar to those used in impregnation of the cable. The modified couplings must be as light as possible and must be substantially of the same diameter as the rest of the cable. The couplings designed by the authors provide hermetic sealing of the compound and meet the above conditions. P. A.

315. Using Alcohol Fuel in Brazil. *Oil and Gas J.*, 1931, 29 (34), 122.

Alcohol prepared from sugar cane has been tried out with success as a fuel for railway motor-cars by the Brazilian Central Railway. The fuel consists of 42° alcohol, with an addition of 5% of ethyl methylene (extracted from the alcohol) and denatured with 0.001% methylene blue. The consumption of alcohol is stated to be 1·1 litres per k.m. as compared with 1 litre when using gasoline. The cost of alcohol is, however, given as half that of gasoline, since it can be made from excess sugar cane production while the gasoline must be imported. C. L. G.

Coal and Shale.

316. Low Temperature Carbonization. *Report of the Fuel Research Board, Mar.* 31, 1930, 43–52.

During the year 1929, tests on the Turner retort at Coalburn, Lanarkshire, and on the Babcock plant of the Newcastle-upon-Tyne Electric Supply Co., Ltd., were carried out.

The former plant worked smoothly but it was not possible to state how far the results were dependent on the method of applying alternating steam pressure. Steam suffers from the disadvantage of high cost and causes difficulty by the large quantity of very dilute ammoniacal liquor produced.

The Babcock plant worked smoothly and the claims regarding throughput and yields were substantiated. This plant has been fully described. (*J. Inst. Elec. Eng.*, 1930, **68**, 205, J. Inst., Fuel, 1930, **3**, 190.)

The Richmond Retorts, which were built to the general design of those developed at the Fuel Research Station, have been described (*J. Inst. Fuel*, 1929, **3**, 18, 125.)

Low temperature tars and spirit have been further studied and a method for the determination of unsaturated, aromatic, and naphthene, constituents has been developed. Cracking the tar by ordinary liquid phase methods is not promising and high pressure cracking in the presence of hydrogen has therefore been carried out in 2-litre bombs similar to those used by Bergius. The variables, temperature, pressure, concentration of hydrogen and effect of other gases, rate of heating and time, have been studied. There is no appreciable change up to 350° C. From 400°–500° C. appreciable cracking takes place, and above 500° C. excessive coke is formed with an initial press of 60 atm., but with 40 atm. some coke is formed. At 400° no coke is formed with an ititial pressure of 20 atm. At 430° no coke is formed. At 450° a small amount of coke is formed with an initial pressure of 80 atm. In general, the higher the temperature the greater the pressure necessary to prevent coking, otherwise the effect of pressure is small.

Experiments are also in progress on the vapour phase cracking of the tar fraction boiling up to 230° C., special attention being paid to the reduction of tar acids and the effect of catalysts.

Diesel and fuel oils have been prepared from low temperature tar and some notes are given on tests made by the Admiralty Experimental Station at Haslar and by the Admiralty Engineering Laboratories at West Drayton, the former using the tar oil as a fuel oil, and the latter using a similar tar oil in a Diesel engine. R. P.

317. Hydrogenation of Coal and Low Temperature Tar. *Report of the Fuel Research Board*, Mar. 21, 1930, 68–69, 103–115.

Experiments with coal have been continued in the intermediate scale continuous one ton per day plant and in small bombs. Attention has been specially directed to the study of hydrogenation of ultra clean coals with exceptionally low ash content. It was found possible to clean Beamshaw Coal from South Yorkshire until the ash content was only 1·3%.

Investigation of the partial hydrogenation of coal, whereby products having much higher coking properties are obtained, has made considerable progress. Considerably larger proportions of the solid products are soluble in pyridine and benzene than is the case with the original coal.

Work upon British coals has confirmed the work of Bergius on Continental coals. In a modified intermittent process a charge of 50 lb. of coal was mixed with 2½ lb. of "luxmasse" and placed in a large

bomb. The bomb was heated to 430° C., and the charge stirred for 5½ hours whilst hydrogen was passed through. The product was condensed from the hydrogen stream and amounted to 69% of the dry ash-free coal. It was unlike any oil previously obtained, as it was clear, transparent, and red brown in colour. (Normally Bergin product is a thick black tarry liquid.) The oil was free from solid matter, and on distillation to 360° left a residue which was not pitch, but a heavy waxy oil.

The oil appeared particularly suitable for further hydrogenation with catalysts, and experiments have yielded encouraging results. These results show that it is possible to obtain about 80 gal. of light oil boiling below 230° C. per ton of coal substance, and there seems no reason why, with reprocessing the heavier oils, yields of 120 to 130 gal. per ton should not be obtained.

Low temperature tar has been treated in two parts, that boiling below 230° C. and that boiling above 230° C.

The best conditions of working were found to be 450° C. and 100 atm. initial pressure of hydrogen. Eighty different catalysts have also been tested and the best general results were obtained by using a mixture of molybdic acid and sulphur, although the highest yields of light spirit were obtained with hydriodic acid. To save hydrogen, the tar acids can be reduced to neutral oils by means of water gas in conjunction with molybdic acid and sulphur, in a first stage and hydrogen can be used in a second stage.

The catalysts show promise of conversion of tar into oil free from pitch and a continuous apparatus has been designed to allow the reaction to proceed further than in the static experiments. In this plant hydrogen and tar will flow counter current in order to make possible the continuous removal of products and maintenance of the concentration of hydrogen. R. P.

318. Investigation of Brown Coal Benzine Produced by Hydrogenation. J. Varga and I. Makray. *Brennstoff-Chem.*, 1931, 12 (2), 21.

Previous investigations of benzines produced by hydrogenation refer mostly to the product from a mixture of coal and residue oil. In this research work brown coal without admixture with oil was hydrogenated in a rotary autoclave under an initial pressure of 110 atms. of hydrogen. The apparatus was heated to 470° C. and maintained at that temperature for 1 to 2 min. Under these conditions 50% of the coal substance was converted into benzol soluble products. The oil produced was separated from water and distilled from an iron still and a fraction boiling up to 230° C. collected.

The raw benzine fraction thus obtained had a sp. gr. of 0·884 at 22° C. and contained 21% of tar acids. The boiling points and phenolic content of the fractions from the raw benzine were:—

Up to 100° C.	13·5%	..	phenols	2·0%
100–150° C.	30·0%	..	,,	6·0%
150–200° C.	35·0%	..	,,	27·5%
200–230° C.	20·1%	..	,,	42·0%

Analysis of the bases soluble in 10% acid, to establish their character, was carried out and the carbolic acid, benzene, toluene, and xylene besides the unsaturated, aromatic and other hydrocarbons were determined.

The bases were removed with 10% sulphuric acid and the phenols with 14% soda lye. The residue was distilled and the unsaturated compounds removed by shaking with 1·84 sp. gr. H_2SO_4, whilst the aromatic content was determined by treatment of the acid and base free oil with 100% H_2SO_4. Deducting the unsaturated absorption as previously determined gave aromatics by difference.

The residue contained the paraffins and hydroaromatics only. The analysis thus far showed—Phenols, 21·0; bases, 3·0; unsaturateds, 6·6; aromatics, 11·5; paraffins and naphthenes, 53·8; loss, 4·1%.

The bases soluble in weak acid were recovered by soda treatment and extraction with ether. To determine their character they were again dissolved in 15% H_2SO_4 and the solution cooled and diazotised. The nitrosamines were extracted with ether leaving the tertiary bases. It was found that the bases were mostly tertiary in character.

After removal of the bases the phenols were dissolved out and fractionated to constant boiling limits.

The benzol, toluol, and xylol were determined, after the bases, phenols, and unsaturated compounds had been removed, by careful fractionation to constant boiling limits then the benzene and toluene were determined by nitration and the xylene by sulphonation.

No naphthalene was found by analysis, although Heyn and Dunkel have stated (*Brennstoff-Chem.*, 1928, 7, 249) that naphthalene was found along with tetraline and decaline in Bergin benzine.

The complete analysis was as follows—Bases, 3·00; phenol, 1·58; cresols, 19·42; unsaturated, 6·60; benzene, 0·70; toluene, 2·02; xylene, 2·65; other aromatics, 6·13; paraffins and naphthenes, 53·80; loss, 4·10%.

R. P.

319. Some Experiences with Blast-Furnace Tar. A. Jaques. *J.S.C.I.*, 1930, **49**, 1094–1095.

Blast-furnace tar (which is essentially a low-temperature tar) in the dry condition is prone to exothermic polymerisation. This was indicated by maintaining two dried samples, one at a temperature of 85° C. and another at 100° C. for a number of days. Viscosity determinations were made daily, showing that the sample maintained at 85° C. had increased in viscosity by approximately ½ at the end of 17 days, whereas the other was 15 times as thick at the end of 7 days.

This change also affects the yield of oil from the tar, the higher temperature treatment giving 43% on distillation up to 320° C., the sample maintained at 85° C. gave 58%, whereas the untreated tar gave 61%, calculated on dry basis. Air blowing during distillation reduced the oil yield to 30–40%.

The replacement of air used for blowing during distillation by steam has been tried out on large-scale plants with success, as 55–60% of oil has been obtained compared with an average yield of 22% from the old stills. The oil thus obtained contains 25–30% of phenolic bodies, whilst the highest boiling fractions set to a solid waxy material. W. A. W.

320. Estonian Shale-Oil Industry. *Chem. Trade J.*, 1931, **88**, 56.

A report from Reval states that negotiations have been concluded for the securing of foreign capital, chiefly from England and Germany, for the expansion of the activities of the Estland Mineral Oil Co. (A. S. Eestikiwioli). This concern has hitherto been almost exclusively en-

gaged in the mining of shale-oil and in the production of crude oils. (In 1929 it produced 26,500 tons of shale and 2280 tons of crude oil.) A certain amount of benzine has been previously prepared from the crude shale-oil, chiefly for export to Czecho-Slovakia as alcohol denaturant. It is stated, however, that as a result of improvements in the process of extraction, benzine will be the chief product. A large electric power plant is being erected on the Narwa Waterfall by the Mineral Oil Electricity Company, to provide sufficient power for the new scheme.

The largest individual undertaking in Estonia is the State Oil Shale Works, which mined 365,700 tons of shale in 1929, yielding 5450 tons crude oil. A large scale attempt was made to place the benzine from this crude on the Reval market in considerable quantities in 1929, but the material did not find much favour on account of its very marked odour, as a result of which it was withdrawn. C. L. G.

321. South African Shale Oil. *Chem. Trade J.*, 1930, 87, 632.

A new company entitled Venezuelan Shale Oil and Mineral Company (capital £15,000) is being formed to extract crude oil from shale in the vicinity of Underberg, Natal. Machinery ordered from Scotland is expected to arrive in April or May. The deposits are stated to be very extensive and to be the richest in South Africa. The company expects to distil 2,400 gallons of oil daily. R. A. E.

322. Preparing Liquid Products From Coal or Coal-containing Materials. H. D. Elkington. E.P. 330,223, 31.12.28.

Liquid products are prepared from coal or carbonaceous materials, by rapid heating with hydrogen, with or without catalysts, under pressure to an elevated maximum temperature, and immediately thereafter discontinuing the heating so that the mass begins to cool. The gaseous products are drawn off while still at a high temperature and cooled to obtain liquid hydrocarbons. The residue in the reaction vessel is further heated with hydrogen at an elevated temperature with or without catalysts for some considerable time, the gaseous products of the reaction being cooled as before to yield hydrocarbons, the non-condensable gases of high calorific value for fuel purposes being separated. The following example illustrates the process—200 g. of brown coal containing 24·1% oxygen in the form of organic compounds and 7% mineral constituents are rapidly heated for about 40 min. in a closed vessel in the presence of a molybdenum containing catalyst together with hydrogen under pressure till the temperature reaches 460° C. The pressure is at that moment nearly 240 kg/cm. Directly afterwards heating is stopped, and when the temperature is approximately 325° C. the converted products are drawn off and condensed. In this manner 15·6% by weight of the original dry and ash free brown coal, of benzine and kerosine is obtained. Furthermore 4·6% phenols, 12·5% water and 23·2% non-condensable gases, which contain a large quantity of totally oxidised compounds (CO_2) are found in the draw-off products. In the reaction vessel there remains 41·0% high boiling asphaltic residue, which is again treated with hydrogen under pressure in the presence of a molybdenum containing catalyst. The mass is heated to a temperature of 470° C. and maintained for fifteen minutes at 480° C. The heating is then

stopped and the products drawn off. A further quantity of 12·0% by weight of benzine and kerosine, and 1·4% phenols is obtained. Further about 4·6% non-condensable gases of high calorific value are obtained. Finally in the reaction vessel there remains 20·4% asphaltic residue which is again heated with hydrogen. This operation yields still 4·4% benzine and kerosine, 0·2% phenols and 14·4% residue, which when distilled under vacuum yields 7·3% thin liquid oil and 7·1% hard asphalt. H. E. T.

323. Destructive Hydrogenation. C. F. R. Harrison, E. D. Kamm Imperial Chemical Industries, Ltd. E.P. 338,576, 16.7.29.

Carbonaceous matter, such as coal or heavy oil, is converted into petrol in two stages. In the first, the material is submitted to hydrogenation whereby mainly middle oils are formed. In the second, the middle oils are converted into petrol by a similar treatment in the vapour phase. Catalysts may be used in both stages.

The liquid phase converter may be a vertical tower maintained at a temperature of 420–450° C. under a pressure of 250 atm. The coal paste may consist of 2 parts of coal to 3 parts of oil, and is preheated nearly to reaction temperature. The quantity of hydrogen supplied may vary from 500 to 2000 cu. m. per ton of paste, and is also preheated if desired to a temperature above the reaction temperature to compensate for heat losses. A catalyst, *e.g.*, iron oxide, may be incorporated in the paste, and a massive catalyst, *e.g.*, tin plate, may be arranged within the converter.

The vapour phase converter may also be a vertical tower containing trays supporting a catalyst such as iron oxide, gels of oxides of tin, cobalt, nickel, iron, or molybdenum. Carbon black impregnated with tin may also be used. Suitable temperatures are from 450–480° C., and pressures may be about 250 atm.

The incoming oil and hydrogen are preheated to near the reaction temperature. A suitable amount of hydrogen is 1000 cu. m. per ton of middle oil.

As an example, a paste consisting of 30% powdered coal and 70% heavy oil was mixed with 2% of Fe_2O_3 and passed through a preheater into the vertical reaction vessel at a rate of 10 kg. per hour, where it was in contact with hydrogen at 420° C. and 250 atm. The hydrogen was preheated and fed at the rate of 10 cu. m./hour (n.p.) whilst the reaction time was 2 hours

The total products were withdrawn and the heavy oil and solids separated in a catch pot at 350° C. This separated mixture was filtered and centrifuged to remove sludge, and the heavy oil, after mixture with fresh coal and catalyst, was returned to the reaction vessel. The low boiling products consisting of 25% petrol and 35% middle oil were passed through a fractionating tower which returned heavy oil to the catch pot, and the vapours were led to a second catch pot where middle oils were condensed at 200–220° C.

This condensate, along with hydrogen at a rate of 2 k.g./hour, was conducted into a second reaction vessel packed with tin plate, maintained at 480° C. and 250 atm., so that hydrogenation took place in the vapour phase. One cu. m. of hydrogen per kg. of oil was passed into the vessel and the contact time was 3 min. The products from this reaction vessel

were conducted back to the catch pot maintained at 200–220° C., and the unconverted middle oil thus returned to the process.

By this process 50% of petrol was produced from coal passed into the liquid phase converter. The middle oil referred to boils between 200 and 300° C. R. P.

324. Utilisation of Gases Produced through Distillation of Similar Treatment of Bituminous Materials. R. Teon. E.P. 338,869, 22.8.29.

The gas-water which is obtained in gas scrubbers of an illuminating gas production plant is heated, and at the same time sulphur purification mass or impure sulphur obtained from the same or a similar source is roasted and burnt in a suitable furnace. The gaseous products which are produced in this way are discharged into a container and brought into mutual contact. This container is supplied with high-tension current of about 70,000–80,000 v., which creates an ionisation of the gases. The products of the reaction of the ionised gases is a solid, fine condensate, which is collected at the bottom of the vessel, and contains all the nitrogen and is perfectly white and pure. To improve the product for manuring purposes it is recrystallised and dried. H. E. T.

325. Destructive Hydrogenation of Carbonaceous Materials. N. V. de Bataafsche Petr. Mij. E.P. 339,875, 13.2.30. Conv. (Holland), 11.3.29.

Favourable results are claimed for the hydrogenation of coal in presence of a catalyst prepared by mixing 44 parts anhydrous ferric chloride and 11 parts aluminium chloride in water and precipitating with ammonia. The product is washed, filtered off and dried at 150° C.

With Carisborg brown coal the yield of products were: water 12·5, benzine 7·9, kerosine 7·2, cresols 4·5, heavy oil 41·9 and coal residue 3·7% of dry and ash-free coal substance.

The process is not restricted to any particular method of performing the destructive hydrogenation. R. P.

326. Distillation of Bituminous Shales, Cannel Coals, Torbanites, etc. F. Esling. E.P. 339,963, 12.9.29.

Powdered shale is mixed with petroleum oil and the mixture subjected to distillation at (*a*) atmospheric pressure, (*b*) slight pressure above atmospheric, or (*c*) vacuum or reduced pressure and at a low temperature, to the necessary degree to drive off a distillate of uncracked oil having a low sulphur content. An uncoked residue is left which is suitable for use in road making and waterproofing. A vegetable oil may be added to the mixture for the purpose of assisting in the elimination of sulphur, and the distillation may be carried out in the presence of steam. W. H. T.

See Abstracts Nos. 268, 269.

ABSTRACTS.

Geology.

327. Time Versus Temperature in Petroleum Generation. P. D. Trask. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 83–84.

In a contribution to the discussion of a recent paper by D. White on this subject, Trask extends the conclusions based on Maier and Zimmerly's work on oil-shale by giving data of his own researches on the effect of heat on modern organic muds. He gives quantitative figures of the amount of organic matter converted into bitumen at temperatures ranging from 280° to 339° C. He concludes that the heat due to blanketing in the earth's crust probably produces bitumen from the organic contents of the sediments and suggests that the formation of bitumen is perhaps an intermediate step in the generation of petroleum. V. C. I.

328. Theories in Origin of Oil Are Questioned. L. G. E. Bignell. *Oil and Gas J.*, 1931, **29** (35), 66. After a paper by F. R. Clark to the Tulsa Geological Society.

Disagreement is expressed with Hager's view, that all oil originates in the widely disseminated matter in shales and limestones, and, that oil can and does migrate long distances in both source and reservoir rocks before being trapped. It is contended that pools occur near or at the place of accumulation of the organic source of the oil. The necessary richness in the organic constituents of the deposit are ascribed to the fact that the majority of oil fields are situated in geosynclinal areas and that the epicontinental deposition then extant was very different from the continental shelf deposition at present in existence, where it is true the organic constituents are sparse and no free oil is found.

It is pointed out that oil accumulations in buried, but truncated structures, are necessarily secondary and probably derived from the cap rock laid unconformably on the eroded surface.

It is also stated that the advocates of the long distance migration theory fail to account for the lack of disseminated oil in the rocks passed through in transit. Laboratory experiments and the difficulty found in Hanover and Pechelbronn, Alsace, of washing sands free of oil by water alone are cited in support of the contention that such disseminated oil should occur.

The localised richness in organic material in the deposits is not put forward as a new theory but as a suggestion for future work in this direction. G. R.

329. Effect of Salt Domes on Accumulation of Petroleum. D. C. Barton. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 61–66.

The author divides the problem of accumulation of petroleum on salt domes into the subordinate problems of:—(1) sources of petroleum, (2) migration of the petroleum, and (3) localisation of accumulation, with its requirements of reservoir space, kind of traps, and water conditions. The source of petroleum has not been shown to have any genetic connection with salt or salt domes.

In the migration of petroleum the effects of salt domes are dual. The increased inclination of the strata accelerates lateral migration and induces convergence of the oil towards the dome; the secondary effect is to facilitate vertical migration through faults and fissures.

The accumulation of oil in the strata above the cap rock is due to simple trapping in an anticlinal structure under hydraulic head. Where the petroleum exists in the cap rock it is due to the porous condition of the limestone and the cover of impervious clays. Where the petroleum exists on the flank of the dome it is due to the existence of such factors as sand lenses, unconformity traps and shale porosity.

The lateral localization of oil around domes is irregular and its causes not well known. Radial faulting, lateral lensing, differences in porosity and local lack of effective sealing are contributory causes. V. C. I.

330. Clay Creek Salt Dome, Washington County, Texas. F. E. Heath, J. A. Walters and W. B. Ferguson. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 43–60.

This salt dome lies in the northern part of the coastal salt-dome province and forms an elliptical dome of about two square miles. Oil was proved in 1928 at a depth of 1,154 ft. and in 1930 the total production was 887,000 brls.

The outcropping formations are basal Miocene and Upper Oligocene and the oil and gas is found in the sands of the Cook Mountain, Mount Selman and Wilcox formations. The careful coring of the wells has proved the presence of important unconformities in the cross section. The author concludes that the upward movement of the dome commenced prior to the deposition of the lower Mount Selman sediments and continued at least to Upper Oligocene times. In addition there has been subsidence in the central portion of the structure, caused by intermittent salt movement over a long period. V. C. I.

331. Seeking for Oil in Southern England. A. Wade. *Oil News*, 1931, **29**, 91–92.

There are plenty of surface indications in England suggesting the presence of oil in the beds below. The conditions of origins, structure, etc., are ideal as regards oil occurrence. A well exists in Derbyshire and showings of oil have been reported in connection with borings in East Anglia, Nottinghamshire, Willesden and the coalfields, but very little enthusiasm has been shown.

The great Wealden anticline of Kent, Sussex and Hants has attracted some consideration of late years. It is an elongated dome with long axis running roughly from west to east 130 miles, and about 40 miles in breadth from north to south, but is not quite symmetrical or uniform. The Weald represents the undulating tract between the North and South Downs. The underlying strata consist of thick beds of clay with thinner partings of sand; below this, sands predominate with smaller beds of clay between the sandy strata. The deposits are Mesozoic and range from Upper Jurassic (Purbeck Beds) through the Lower Cretaceous to the Chalk. The strata are highly fossiliferous and contain evidence to show that they were laid down in estuarine conditions, in a sub-tropical climate. These conditions are very favourable to oil occurrence.

Gas has been struck at 200 and 300 ft. at Heathfield in Sussex and has been used for lighting purposes. A further boring gave more gas at 377 ft.

Some believe that oil will be found in the Jurassic strata under the main fold, while others believe that the chief discoveries will be made in the Carboniferous or even older strata below. The seeker for oil must decide who he is going to follow. The Mesozoic beds underlying the Weald are sufficiently interesting as regards possible oil accumulation to justify closer attention and even the sinking of boreholes to test the strata. E. B. E.

332. Geology of Oil Fields of Polish Carpathian Mountains. H. de Cizancourt. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1–39.

This paper is an attempt to investigate the factors which govern the distribution of the Polish oilfields along the Carpathian Mountain front. After describing the stratigraphy of the oilfield strata, ranging from the Cretaceous to the Miocene, the author discusses the tectonics and relations of the oil pools to the folding and thrusting. The oil is found in two distinct tectonic belts, (1) The marginal belt, as at Boryslaw, associated with the great Carpathian frontal thrusts, and (2) the central depression where the folding is sharp and the oilpools are situated on definite anticlines.

The main oilfields on the marginal zone do not coincide everywhere with the uplifts on the over-thrusts and there is some discrepancy between the surface structures and the present position of the oilfields. The author concludes, therefore, that the oilfields were determined by the facies of the deposits and by early folding movements which preceded the great over-thrusts. V. C. I.

333. East Texas Cross Section Offers Interesting Study. C. F. Dally. *Oil Weekly*, 1931, **60** (6), 32.

Recent discoveries in Rusk and Gregg Counties have resulted in the opening-up of a vast area in East Texas, hitherto neglected.

The East Texas Basin is an embayment having a maximum depth of 5 miles below sea-level, attained in the Sigsbee Deep. Bounding this basin on the west and north is the Mexia Fault, and on the east the Sabine uplift; no southern boundary can as yet be fixed.

The age of the oil-sand is uncertain, opinions being divided between Woodbine and Tokio. The author considers this question can be settled by a study of stratigraphical cross sections, and in this connexion submits one phase of relationship of the Woodbine Sand to the producing horizons in the Joiner and Bateman areas.

The sub-surface structure of the East Texas Basin can at present only be generally outlined, the drilling of additional wells being necessary before this can be proved. As a result of geological mapping in the Gregg-Rusk region, however, two possibilities present themselves (1) that a long pool may arise along the flank of the Sabine uplift, and (2) the area may develop into a series of separate pools at intervals along the flank of the uplift. G. S. S.

334. Investigating the Geological Structure of the Sub-soil. R. Ambronn. E.P. 339,964, 12.9.29.

This invention comprises A.C. generating plant for feeding sub-soil with electric energy, for electric earth investigations, comprising generating means for at least two phase displaced current systems of equal frequency. H. E. T.

Field Technology.

335. Methods of Increasing the Yield from Oil Sands with Special Reference to Repressuring. L. C. Stevens. *J. Inst. Petr. Techn.*, 1931, **17**, 73–101.

Various methods have been tried in recent years with a view to increasing the percentage yield from an oil sand, and these methods are dealt with in some detail in this paper. An examination of the conditions existing in an oil sand is made, and the results obtained are carefully applied to the process

of repressuring. Efficiency of control is essential if the maximum yield is to be obtained. Even with careful control the reservoir pressure falls below the level when producing gas lift or pumping is economical and the process of repressuring must be resorted to. The ideal state would appear to be achieved when the gas is returned to the sands during the flush period, and the pressure maintained as nearly as possible at reservoir pressure.

The author has dealt with the repressuring of the flush and semi-flush fields, and also with the exhausted fields, and gives in some detail the various conditions that must be satisfied in order to obtain the ideal sites for the input wells. A study of the plants used and the methods of collecting and distributing the gas is also made. The paper is a collection of all the available data on the subject of repressuring, and is presented in the form of a review.

The author arrives at the following conclusions for obtaining the greatest possible recovery of oil from any sand with minimum production costs:— 1. Careful control to give minimum gas-oil ratio is essential. 2. Repressuring operations should be commenced during early life of field. 3. Repressuring must be resorted to if the gas energy of the field has been allowed to dissipate. 4. Water drive is permissible if the above method is unsuccessful. Some system of unit operation or co-operation is essential if the maximum benefit is to be obtained. L. W. C.

336. Separation of Sand Carried over by the Muddy Liquor in Hydraulic Drilling. E. Fischer, *Mon. Petr. Roumain*, 1931, **31**, 65–66.

The proportion of sand in a good muddy liquor used in rotary drilling should be about 5–10%, but this is increased as the drill passes sandy formations, and an installation for removing the excess sand while the liquor is in continuous circulation to and from the well is described. Removal of the sand facilitates pumping and also enables mud of any required density to be obtained, while in addition the cost of drilling is appreciably reduced.

The liquor from the well enters a receiving tank with a mechanical agitator, from which it passes to a Merco centrifugal separator. The mud goes on to the storage tank which supplies the well, while the sand which settles to the bottom passes to a dump. An arrangement is made by which, if necessary, this sand can pass to a settling tank divided into compartments from which crude petroleum carried over by any petroliferous sand is removed. This is both an economy and also a safeguard against fire. This settling tank can be used independently of the sand separation installation to separate the crude petroleum from other productive wells. C. C.

337. Casing [Wells] with "Single Column." E. Fischer. *Mon. Petr. Roumain*, 1931, **31**, 66–68.

Two wells are to be drilled, one at Boldesti and the other at Moreni-Tzuicani, in each case using as an experiment "single-column" casing.

It is claimed that, by cementing, the beds of crude petroleum, gas and water are completely isolated from each other behind the same column. Flooding of the petroleum and gas beds has been eliminated, while, at the same time, the details of drilling have been simplified. The following points must be considered, however: 1. The depth to which the hole can be drilled without casing. This depends on the diameter of the hole, which should not exceed 350 mm. Technical considerations may necessitate casing the first 200 metres with a separate column (but this may be withdrawn after cementing). 2. The diameter required for the efficient exploitation of the petroliferous beds. The minimum is given as 5 ins. 3. The ability of the

tubes to withstand the various pressures to which they may be subjected. This factor together with the depth of the well limits the diameter of the column. Below 1000 metres, reinforcing by cement has been found satisfactory. 4. The height at which cementing can be carried out. This depends on the properties of the cement.

Relative costs are given in which it is shown that the cost by the single column system is about ½ that of the old method and ¼ that of the modern method. An estimate is given that about 500 million lei would have been saved if this method had been adopted for the wells drilled during the last two years on three fields. C. C.

338. Some Aspects of Modern Oilfield Practice. C. T. Barber. *Rec. Geol. Surv. India*, 1930, **63**, Pt. 4, 379–427.

This paper deals with two important aspects of modern oilfield practice—viz., the rotary system of drilling and the recovery of oil. The author summarises existing knowledge on these subjects, outlines the extent to which they have been successfully applied in the United States, and discusses the possibility of their application in Burma.

The main advantages and disadvantages of the rotary method compared with the cable tool system are dealt with. The important functions of mud-laden fluid is fully discussed, and reference is made to the successful results obtained in various American oilfields. These include Garber, Tonkawa, Santa Fé, and Midway Sunset. Mention is also made of the advantage of rotary practice, in that the control of high-pressure oil and gas sands during drilling is made possible. The disadvantages of the practice are inaccuracy of drill logs, contamination by mud fluid of low-pressure producing formations, and deviation of drill holes. Contamination by mud fluid has resulted in the control and restriction of the rotary practice in the reserves of Yenangyaung. In loose sands the fluid has been known to penetrate 1000 ft., and in the upper depleted zones of these reserves it was sufficient to mud up producing wells in the district, and production in some instances was never recovered. Deviation of rotary drill holes is discussed, and a table given showing drift and inclination of oil wells with data from about 250 drilled holes in California.

Attention is given to the factors controlling production, the most important being the proper control of pressure and the efficient use of the original gas content of the reservoir. Theoretical considerations are given to the latter, and results are quoted from various fields.

Secondary methods are discussed—viz., those of production under vacuum and repressuring, and it is shown in field practice that the latter is superior to the use of vacuum. This is exemplified by changes in practice in the Mid-Continent area, where during the past two or three years repressuring has largely taken its place.

Conditions in the Burma oilfields (apart from the Twingon and Beme reserves) seem to be such that large increases may be expected by repressuring. The Burmah Oil Co. have already experimented with this method in the Yenangyaung field with encouraging results, and activities are likely to increase considerably along these lines in areas operated by that Company. G. S. S.

339. Operation of Surge Chambers on Pipe-Line Pumps. W. R. Reed. *Petr. Eng.*, 1930, **2** (4), 169–170.

The surge chamber is attached by suitable means to the discharge line of a reciprocating pump, and is used for absorbing shocks caused by the irregular pump discharge. Where two or more pumps are discharged into the same

line or lines this shock is much more pronounced. The gate valve below the surge chamber is opened slightly to allow fluid to fill up to the liquid level, and at the same time a bypass valve is opened allowing the fluid to pass around the piston. These valves are then closed, and the portion above the liquid level is then filled with compressed air to approximately the pressure at which the line is to operate. As soon as the pump is started and the pressure is up to normal, the gate valve can be opened wide and the bypass valve closed. The air cushion then absorbs all the shocks instead of the latter being transmitted to the various parts of the pumps.

It has been found that shock pressures are sometimes three times the average line pressure carried, resulting in pump and line breakages. The expansion of the air in the top of the chamber above the liquid drives the piston downward, and the liquid below the piston being trapped allows the piston to come to rest gradually.

To start up, the gate valve is opened, and as soon as the pressure in the line exceeds the pressure in the air trapped above the piston, the automatic cut-in valve allows the pressure to build up underneath the piston, lifting the entire assembly off the seat. When the valve has sufficiently cleared the seat, allowing the fluid to pass, the cut-in valve closes due to spring tension. The piston continues to be carried upward as the line pressure increases until the air above is compressed to a pressure approximately equal to the pressure below. Any shocks are then absorbed by the trapped air. L. W. C.

340. Use of Indicator Diagrams in Checking Engine Results. H. V. Beck. *Petr. Engr.*, 1930, **2** (4), 73.

Indicator diagrams are recognised as a means of checking engine performance in gas compressor stations, but their full significance as indicators of the condition of the compressor is not commonly understood. A graphical representation of the pressure conditions in a closed cylinder is obtained from the indicator diagram, the vertical and horizontal components being proportional to the pressure and piston displacement respectively. Remembering this, the analysis of an indicator diagram becomes a simple matter.

A number of compressor diagrams are given and examined to discover the various defects in the running conditions. Where the engine speed is governed by throttling of the fuel gas, lean mixtures may result from light loads resulting in curves very similar in appearance to those obtained by a late spark. Full load must therefore be put on the engine when observing the ignition. In the equation to find the indicated horse-power the factor which may cause difficulty is the mean effective pressure. Sample calculations are given with a view to simplifying the method of procedure. L. W. C.

341. Mud Fluid is an Important Factor in Deep Drilling. W. A. Sawdon, *Oil Bulletin*, 1931, **17**, 127.

Weight of mud fluid must naturally be considered, but there are other functions the mud must perform both before and after penetration of the productive horizon. These functions increase in importance as the depth increases, and are dependent upon the colloidal properties of the solid matter contained. Although there is some relation between colloidal properties and viscosity a high viscosity does not necessarily indicate high colloidal properties. The viscosity of a mud fluid is a difficult quality to determine, since the fluid is not strictly a fluid. A negligible amount only of solid matter is in true solution, the remainder being in suspension. The suspension, however, may be regarded as complete as far as the time required for drilling operations is concerned.

When determining the viscosity of the fluid, these solids must not be considered in solution. A mud fluid composed of a large amount of quickly settling solid matter may have a viscosity so high that the pumps will not handle it, and yet have little or no colloidal properties. These colloidal properties are essential for good rotary drilling mud, and without them many of the necessary functions cannot be obtained.

Admixtures have been used with considerable effect to increase both the weight and colloidal properties in the mud fluid, but their general use can only come with greater knowledge.

Viscosity and colloidal qualities are necessary to accomplish one of the primary uses of rotary drilling mud—that of removing the cuttings from the bottom of the hole, and the use of an admixture appears to be the best method of regulating the fluid to meet these requirements.

The lubricating and sealing properties of a mud have always been considered important, but in the deep wells much more importance should be attached to their presence. Both these qualities are dependent upon the colloidal matter. The lubricating quality facilitates running casing and reduces friction in the drill-pipe and decreases the wear on the pumps.

The addition of some colloidal matter is, in general, necessary, and its introduction into the well should be done in a proper manner. Various methods are adopted, the most general being by means of a small hopper. It is not to be expected that weighting admixtures will also add colloidal qualities to the fluid. For a perfect admixture they should do so, but their chief function is to increase the weight of the mud fluid. Control of drilling fluid is an important factor, and great care should be taken to see that the admixture is added in the proper amounts. Incorrect proportions of admixtures to mud fluid may result in failures. L. W. C.

342. Drilling in Pacific Requires Substantial Foundation. L. B. Collins. *Oil Weekly*, **60**, 24.

The foundation structures necessary for derricks erected in bodies of water require to be stronger than for land locations, and a stabilised foundation construction has been developed. The old method was to erect several columns of concrete in the ocean bed joined on top into a table upon which the rig was set. This resulted in a large total perimeter of coffer dams. The expense of excavation to penetrate the shales and the necessary bracing in this type of construction was extremely high.

The new type of foundation consists of three essentials: (1) A central column member; (2) stabiliser around the column at ocean bottom; and (3) foundation top on column. The central column affords ample resistance to bending with a minimum area of exposure to wave action. By proportioning the stabiliser to suit the depth of water and bottom material encountered a horizontal thrust at ocean bottom level is furnished without creating the waves unduly. The foundation top on the column allows a 26-ft. square for the erection of a standard steel derrick, and the rotary table is connected to the column with ample strength to meet all stresses of the deepest rotary drilling operations. The central column coffer dam, composed of sheet steel in the shape of a 19 ft. diam. circle, is driven first. Stiffeners are bolted to the interior. The sea bottom is then excavated until a hard shale is met, and 32-in. conductor pipe is then set. The stiffeners are then removed and placed in a 15-ft. diam. circle to form the vertical reinforcement. Horizontal reinforcements are then placed between the vertical members and the sheet piling, and concrete then poured in through a Tremie tube.

The stabiliser must next be set, and consists of a steel cylindor ¾ in. thick reinforced with angle-iron ribs, and is about 40 ft. in diam. and 15 ft. high. This is lowered over the central column and is set at a fixed depth in the ocean bed. Steel piling is then driven around the inside edge of the ring. Reinforcing is then lowered into place and concreting done in the same way as for the column. L. W. C.

343. D.C. Electric Drive for Portable Drilling. W. A. Sawdon *World Petr.*, 1931, **2**, 157–159.

Where it is possible to deliver power to wells from a central power plant, alternating current lends itself to more economical generation, distribution and utilisation. Low first cost and simplicity are factors which have assisted in its development. Where new fields are being opened up steam and gas engines may be unsuitable and the utilisation of alternating current impracticable. For this reason an electric motor drive with provision for the generation of direct current at the well has been developed.

The d.c. motor is much more flexible than the a.c. type, and its performance approximates that of the steam engine. It provides a high starting torque, a high efficiency under load, and accelerates smoothly. The d.c. generating plant consists of two units. Two 125-K.W. d.c. generators driven by two 200-h.p. 6-cyl. Diesels. An exciter is mounted on the same shaft as the generator and provides current for lights and auxiliary power. The motors are totally enclosed and of the spark-proof type. Control is obtained by a push button at the drilling position, and the generators can be placed in series by means of a switch, so that double voltage and power can be given to drilling motor with the pump cut out. Greater portability is obtained by using gasoline engines in place of the Diesels. Two units are used, each consisting of a 165-h.p. gasoline engine coupled to a 75 K.W. d.c. generator and an exciter. The two units can be run in parallel or separately. With the portable unit an automatic weight control has been developed, and the whole unit can be easily transported on trucks. L. W. C.

344. Core Drill. W. H. Harvey. U.S.P. 1,790,543, 27.1.31. Appl., 6.3.29.

This invention consists of a drill collar, the bore of which is flared at its lower end, a core drill carried by this collar, and a core barrel for receiving the core. Placed longitudinally on the outside of the core barrel are a number of flexible arms which normally bind against the walls of the collar. At the lower ends of these arms is a cutting element which is turned inwards. Upon raising the core barrel the arms will be forced inwards over the lower end of the core barrel as the arms ride over the walls of the bore. L. W. C.

345. Drilling Device. A. M. Gildersleeve and C. L. Dean. U.S.P. 1,790,613, 27.1.31. Appl., 10.11.26.

This invention consists of a bit body into which are mounted a number of rotatable cutters of frusto-conical shape. These cutters are so placed that they cut at angles substantially less than 90°, and are in fixed relation to each other. They rotate about their respective axes due to being skewed relative to their direction of travel and to their being located at different elevations and at different distances from the centre of the bit. These cutters, therefore, cut in a number of circular paths of varying radii, which effectively embrace the area to be drilled at the bottom of the hole. The lateral thrust of one cutter is balanced by the other cutters travelling in different paths due to their arrangement. L. W. C.

See also Abstract No. 387.

Crude Oil.

346. Continuous Distillation of Mineral Oils. Soc. pour l'Exploitn. des Procedes Ab-der-Halden. E.P. 340,370, 11.11.29.

Mineral oil is preheated in an apparatus with an extended heating surface. The hot oil is introduced, together with superheated steam, into a distilling receptacle of large capacity. The preheating of the oil, the superheating of the steam and the heating of the distilling receptacle are all effected by means of one furnace. W. H. T.

347. Treating Oil. J. Delattre-Seguy, Assr. Universal Oil Products Co. U.S.P. 1,782,809, 25.11.30. Appl., 28.6.26.

This apparatus consists of a brick combustion chamber the lower side of which may be a brick wall or a metal plate through which heat radiates into the heating chamber situated below. In this chamber an inclined, wide, shallow channel is mounted on adjustable supports so that its slope may be varied at will. Oil is introduced at the upper end of this channel through a tranverse, perforated nozzle and the residue from the lower end is collected in a wide funnel connected to the residue line. The vapours generated pass out through a vapour collector to any suitable condenser and receiver. W. L.

348. Treating Hydrocarbons. J. Delattre-Seguy, Assr. Universal Oil Products Co. U.S.P. 1,782,811, 25.11.30. Appl., 28.6.26.

This apparatus consists of a shell still mounted in a heating chamber. This shell has all except the upper portion of its inner wall lined with firebrick, while the upper portion serves as a radiating surface through which the heat of combustion passes into the still. Inside the still is mounted an inclined wide shallow channel the slope of which can be varied at will. Oil is fed to this channel at the higher end through a transverse perforated nozzle and the residue is drawn off from the bottom through a wide funnel connecting with a residue line. The shell is provided with a vapour pipe and the vapours are led to dephlegmators or direct to condensers as desired.

The apparatus may be modified by dispensing with the heating chamber and supplying the necessary heat by passing a hot molten material such as molten lead or glass down an inclined trough placed above the oil channel. This trough runs longitudinally through the still above the oil channel but is inclined in the opposite direction to the latter. W. L.

349. Distillation of Hydrocarbon Oils. G. W. Watts, P. L. Krauel and T. E. Stockdale. U.S.P. 1,784,561, 9.12.30. Appl., 3.4.25.

The invention is more particularly intended for use in connection with the distillation of heavy oils under vacuum. Hot residue oil from a continuous topping battery is brought in contact with superheated steam at a temperature not greater than 900° F. through an expanding nozzle. A deflector is placed immediately in the path of the oil and steam issuing from the nozzle. The mixture of vapours and oil pass to a horizontal cylindrical separator from which any unevaporated oil is withdrawn. The vapours pass from thence to a condensing chamber where the oil is condensed and flows through a vertical pipe to a receiver. Water and any oil vapours escaping from the condensing chamber are dealt with in a water-cooled worm condenser connected with a second receiver. The whole system is closed and can be operated in vacuo. R. W. L. C.

350. Refining Hydrocarbons. E. W. Isom and E. B. Phillips, Assrs. to Sinclair Refining Co. U.S.P. 1,786,493, 30.12.30. Appl., 27.3.29.

The raw material is mixed with an absorbent refining agent, such as "terrana earth," in the proportion of 5% by weight and the mixture is fed to a conventional pipe still where it is raised to a temperature not exceeding 750° F. during a passage period not exceeding 15 min. The hot mixture is discharged from the pipe still into a vaporizing and digesting chamber at a rate sufficient to maintain a liquid temperature of about 620° F. The solid refining agent is maintained in suspension by introduction of superheated steam and by mechanical agitation. The rate of operation is such that the residual product remains in the vaporizing and digesting chamber for a period averaging 100–300 min. The vapours from this chamber are subjected to reflux fractionation in a bubble-cup tower regulated so that the residue discharged from the system has at least the desired flash-point and viscosity of the finished product. For instance, a bright stock residue of a viscosity of 120 at 210° F. (Saybolt) and of 6 colour can be thus obtained in about 21% yield from a Pennsylvania crude petroleum. Cuts of lower viscosity can be obtained as side-drawn streams from the fractionating tower. R. W. L. C.

351. Crude Oil. K. W. Hickey. U.S.P. 1,789,233, 13.1.31. Appl., 11.1.29.

Basic sediment from crude oil emulsion or bad oil collecting at the bottom of tanks, reservoirs, etc., is separated by adding a solution of sodium bicarbonate and borax. Hydrochloric acid is added separately and simultaneously in order to produce a violent reaction and precipitation of the foreign matter and water. W. S. E. C.

Natural Gas.

352. Type of Absorption Oils Dependent on Condition at Plant. S. J. Hunter. *Oil & Gas J.*, 1931, **29** (36), 105.

In the process for recovering gasoline from natural gasoline by oil absorption, the character of the oil has an important influence on the design of the plant and on the recovery efficiency. The oils actually available can be divided into three classes as follows: 1. Light oils, b.pt., 350/475° F.; gr., 38/42° A.P.I.; mol. wt., 160/180. 2. Medium oils, b.pt., 400/600° F.; gr., 36/40° A.P.I.; mol. wt., 180/220. 3. Heavy oils, b.pt., 500/750° F.; gr., 32/39° A.P.I.; mol. wt., 220/275.

The light oils tend to atomise more easily, thus increasing the absorption area, and it is estimated that the absorption factor increases 2%/° A.P.I. rise. Other advantages of light oils are low viscosity, mol. wt. and sp. gr., whilst their disadvantages are high make-up and increased stripping difficulty. The reverse is true for heavy oils and the medium oils are intermediate in character. To show the effect of the type of oil used it is stated that a plant designed to treat a given volume of gas at a certain oil/gas ratio using oil of 180 mol. wt. would be 25% overloaded if oil of 260 mol. wt. were used.

Plants possessing good fractionating apparatus can use an oil having an I.B.P. only 25° F. above the F.B.P. of the finished product and the economies in heat exchange and cooling surface, and also in steam, generally justify the extra cost of such improved plant.

Absorption efficiency is also increased by decrease in temperature to an estimated extent of 2%/° F. and by rise in pressure. Temperature is more easily controlled, consequently some plants are already operating with coolers on the bottom trays of the absorbers, and possibly cooling will be extended to all trays in future plants.

It is generally agreed that unsaturateds are least resistant to oxidation which causes deposition of sludge in the plant, and lengthening of the distillation range of the oil. For efficient operation the maintenance of oil at as near its original condition as possible, is one of the most important factors.

R. A. E.

353. Removal of Sulphuretted Hydrogen from Gases. C. H. Hansen, Assr. to Heinrich Koppers A.-G. E.P. 340,663, 21.10.29. Conv. (Ger.), 22.11.28.

The invention is based on the discovery that iron-containing thionate liquors are capable of bringing about complete elimination of H_2S, irrespective of whether NH_3 is present in the gas or not. The liquors are maintained in a form inhibitive to the formation of FeS by the presence in the scrubbing liquor of polythionates in forms higher than the tri-thionate (*e.g.*, addition of H_2SO_3). The used scrubbing liquor is treated with sulphur containing gases in order to set free iron sulphide, ammonium sulphate and sulphur. The iron sulphide is dissolved (in H_2SO_3) and reused cyclically. An apparatus for carrying out the process is described.

W. H. T.

354. Recovering Gasoline from Natural Gas. N. E. Loomis, Assr. to Standard Oil Development Co. U.S.P. 1,789,470, 20.1.31. Appl., 25.11.25.

The gases and the absorption liquid, which is non-volatile under the conditions imposed, are brought into intimate contact in a tower. The charged absorption liquid is preheated and introduced into an intermediate portion of the vapour and liquid contact zone and passed downwards. The lower portion of the zone is heated and the upper portion constitutes the cooling zone. A means is provided for removing residual gas and absorption oil containing the desired product. This absorption oil containing the desired product is passed to a stripping still and the stripped oil is conveyed from the still in indirect heat exchange with the absorption stock passing to the tower.

W. S. E. C.

355. Extracting Benzene Hydrocarbons from Gases. E. Breitbach, Assr. to K. Still (Germany), U.S.P. 1,790,571, 27.1.31. Appl., 21.11.28.

Benzene hydrocarbons are extracted from gases by means of wash oil containing naphthalene. The benzene charged wash oil is then distilled to obtain a distillate containing naphthalene. The distillate is continuously rectified, the liquid naphthalene-charged residue being passed while still warm to the main stream of wash oil circulating through the gas scrubbing plant.

W. H. T.

See also Abstract No. 425.

Motor Fuels.

356. Detonation of Gasoline. N. A. Butkov. *Trans. Thermo-Tech. Inst. Russia*, 1930 (2), 41.

A bomb (650 c.c.) fitted with a pressure gauge was charged with 2 c.c. of fuel and 5 atm. O_2 and heated at 250° for 90 min. It was found that substances more resistant to detonation were also more resistant to oxidation. C_7H_{16} and $C_{10}H_{22}$ were oxidised to a great extent without an explosion and formed a large amount of carbonaceous material insoluble in C_6H_6. The same change occurred with ethyl ether, CS_2 and pinene. A 10% addition of toluene to C_7H_{16} caused an explosion, this indicating a change in the character of the oxidation; solid products and acids were not formed. Alcohol has a similar effect. The above conditions were identical with those existing in an engine with a 5:1 c.r. Experiments carried out at 285°

made it possible to obtain a finer classification of antidetonant. The properties of fuels were further characterised by the composition of the combustion gases, proportion of oxygen and fuel and other reaction products left in the bomb. W. H. T.

357. Treating Pressure Distillate with Cupric Solution. R. H. Cook. *Petr. Eng.*, **2** (4), 163-165.

The advantages claimed for the method of treating pressure distillate by the so-called "blue solution" method are good colour, stability, reduced sulphur and gum contents and obviation of further treatment of the finished gasoline. The blue solution is prepared by pumping 500 gal. of water into a vat, adding 450 lbs. of copper sulphate and boiling with steam until solution is complete. About 420 gal. of 13·5° Bé doctor solution is added and the whole transferred to a mixing tank and agitated mechanically; 70 gal. of 26°Bé ammonia solution is then added and further agitation given. The mixture contains a precipitate which must be kept in suspension during use. Blue solution is more stable but less effective in cold weather, and must not be kept longer than 20 days or oxidation will destroy its effectiveness.

The pressure distillate is first treated with the required amount of 60·5°Bé acid, which is a mixture of reclaimed and fresh acids. After thorough removal of acid sludge by settling and washing, the distillate is treated with sufficient blue solution to ensure a doctor-sweet product. The whole operation is carried out on a continuous basis, the quantities of the reacting products being carefully metered and acid sludge, spent blue solution and water removed from the bottom of the respective treating vessels. The quantities of acid vary from 2 to 4 lbs. per brl., and of blue solution from 1 to 4 lbs. per brl., according to the nature of the distillate undergoing treatment.

Blue solution is slower in action than plumbite solution, consequently it is beneficial to store the treated distillate from 5 to 15 days before rerunning.

Generally speaking the rerun gasoline is doctor-sweet, non-corrosive, and has a colour of 25–30 Saybolt, depending on the amount of acid used. Recovery of the copper salts is possible on the large scale, but is not practised in the plant described. A flow chart and details of the plant equipment are given. R. A. E.

358. Gas Fuels for Airships. P. L. Teed. *Aircraft Eng.* 1931, **3**, 41–42.

Experiments have been carried out in Germany with a view to obtaining a gas fuel suitable for use in airship engines.

As the production of any single gas presented many difficulties, it was considered that a more satisfactory fuel could be prepared from petroleum. "Blau Gas," after experiment, was found to be quite satisfactory.

The first stage in its manufacture consists in the production of Pintsch gas, by cracking a mineral oil such as "Mineral Seal," Scotch shale, or Russian "Solar" at a temperature of 600–700°C.

A typical plant is described and illustrated diagrammatically.

The resultant gas, purified by lime from carbon dioxide and sulphur compounds, is compressed to about 10 atm., when a small amount of heavier hydrocarbons separate out.

On compressing Pintsch gas to 100 atm. and cooling to normal temperatures a liquid consisting of ethane, butane, iso-butane, propane, pentane, propylene and butylene with ethylene and methane in solution is obtained.

This liquid is "Blau Gas," and it contains about 39% paraffins. This fuel will stand an H.U.C.R. of 9:1.

In the U.S.A., suitable fuel can be prepared from natural gas after extraction of the petrol content by methods analogous to the preparation of "Blau Gas."

Under the name "Pyrofax"—liquefied propane containing 2% butane is marketed. The Graf Zeppelin used this mixed with hydrogen for part of its flight, and mixed with natural gas for another section.

A further alternative suggested is the heavier gases, which could be obtained from the waste gases in liquid or vapour phase cracking of crude petroleum.

C. C.

359. Coal Gas as Motor Fuel. (From information supplied by M. L. Bacqueyrisse). *Gas World*, 1931, **94**, 64.

Experiments are being carried out in France on motor lorries and 'buses, particularly by the Société du Gaz de Paris and the S.T.C.R.P. The vehicles are fitted with the usual 4-cyl. engines, the bore, however, being increased from 105 to 110 mm. and the compression ratio raised from 4·25 to 5·4 to 1.

The gas is carried in cylinders and passes to an expander where the pressure is reduced to slightly higher than atmospheric. From the expander the gas is drawn by engine suction to an automatic gas and air mixer, and thence to one branch of the main inlet pipe. The latter has two branches, the second being connected to an ordinary liquid fuel carburettor; the engine can therefore be run on one or the other fuel.

The daily procedure is for vehicles to obtain their charge of compressed gas on which they run until the gas is exhausted, at which stage liquid fuel consisting of 60% benzol and 40% commercial petrol is used.

The brief comparison of fuel consumption given is: Gas, 48·5 cu. ft. or 6169 cal. per vehicle mile; petrol-benzol mixture, 1·286 pints or 6,054 cal. per mile. The distance run on one charge of gas was found to be up to 50 miles.

W. A. W.

360. Fuels for Internal Combustion Engines. R. T. Hurley. E.P. 315,706, 3.7.29.

Anti-knock motor spirit is produced by the addition to petrol of iron carbonyl in the presence of lead tetraethyl or of nickel carbonyl. The amounts of lead or nickel compound are not to exceed 0·1% of the iron carbonyl present. It is claimed that the effect of the iron carbonyl on the insulation of sparking plugs is thereby counteracted; at the same time the concentration of the poisonous lead or nickel compound is too small to be dangerous.

G. B. M.

361. Mixtures for Addition to Motor Fuels to Reduce "Knock." J. Y. Johnson. E.P. 339,637, 10.7.29.

The use of iron carbonyl as a dope for motor-spirit is complicated by the tendency of this compound to ignite spontaneously even at low temperatures. It has been found that the addition of appreciable amounts of certain aromatic or hydroaromatic amines, alkylated or arylated on the nitrogen atom, renders the iron carbonyl safe for handling and storage.

This patent covers the use of such blended dopes, containing not less than 10% of iron carbonyl. The addition of lubricating oil to the dope, for upper cylinder lubrication, and of colouring materials to facilitate blending, is also covered.

G. B. M.

362. Vacuum Distillation of Hydrocarbon Oils. H. C. Wiess, Assr. to Standard Oil Development Company. U.S.P. 1,786,639, 30.12.30. Appl., 25.9.25.

Oil to be distilled is heated to the distillation temperature (*e.g.*, 625° F.) and the pressure on the oil is reduced to 25 or 50 mm. absolute or less. Naphtha, which has a boiling range of almost 350–425° F. and a vapour pressure of less than that of water at the condensing temperature, is injected into the hot oil. The oil and naphtha vapours are led off to a partial condenser where the oil is condensed. The naphtha vapours are condensed in a final condenser.

R. W. L. C.

363. Polymerising Hydrocarbon Distillates. R. C. Osterstrom, J. Hyman and C. R. Wagner, Assrs. to Pure Oil Co. U.S.P. 1,789,413, 20.1.31. Appl., 25.1.30.

Gum forming compounds are removed from cracked distillates by vaporising them under pressure in the presence of polymerised compounds containing sodium sulphonates. The volatile fractions are separated and condensed, while the heavy polymer-containing fraction is treated successively with acid and alkali in order to secure a solution of sodium sulphonates in polymerised oils. The solution is returned to the polymerising zone for admixture with the cracked distillates undergoing treatment. W. H. T.

See also Abstract No. 365.

Kerosine.

364. Oxidisability of Mineral Oils. B. G. Tuichinin and K. I. Ivanov. *Neft. Khoz.*, 1930, **18**, 979-991.

Baku gas oil when treated with 12% of sulphuric acid (monohydrate) added in three portions, and followed by a washing with tap water, can be oxidised with oxygen at 15 atm. by heating at 150° for three hours to give a saponification number of 104·3. A treatment with 6% of oleum (10% SO_3) under identical conditions yields an oil with a saponification number of 63 after the above oxidation test. An additional treatment of the oil with 50% alcohol solution and washing with tap water increases the oxidisability considerably. If the treated oil still contains small quantities of naphthenic acids it should be washed with 50% alcohol and water. A treatment with 253% of the amount by weight of sulphur dioxide and evaporation of the sulphur dioxide from the oil gives a saponification number of 112·6 after oxidation. The addition of naphthenic acid salts of K, Na, Li, Fe and Mn to an acid-treated oil increases its oxidisability. The above oxidation method is used in determining the stability of oils. W. S. E. C.

365. Treating of Sulphur Containing Oils. H. G. M. Fischer and W. J. Addems. Assrs. to Standard Oil Development Co. U.S.P. 1,789,335. 20.1.31. Appl., 8.5.26.

Sour petroleum distillates containing mercaptans are mixed with preformed lead sulphide, alkali and sulphur in amount equal to one-half of the sulphur content of the mercaptans and the mixture is blown with air. Polysulphides are formed. The distillate is distilled at a temperature not above 320°F. and the sulphur-free distillate is given a final light treatment with doctor solution. W. S. E. C.

See also Abstracts Nos. 405, 414.

Lubricants and Wax.

366. Use of Filter Aid in Dewaxing Lubricating Stocks. *Petr. Eng.*, 1931, **2** (4), 108–110.

At least three commercial plants for dewaxing lubricating oils by the Weir process are now in operation, apparently with varying success. In this process a filter aid is added to a chilled solution of oil in naphtha, and the whole mixture filtered. An apparatus for determining the best operating

conditions for the process comprises a solution chilling tank of at least half-barrel capacity, insulated and provided with an agitator; a naphtha chilling tank of similar capacity; a filter leaf of 0·7 to 1 sq. ft. total surface with connection for vacuum; a graduated filtrate receiver of 1000 to 3000 c.c. capacity, and a vacuum pump. Recommendations as to the most suitable types of apparatus are made.

As an initial guide, the percentages of naphtha generally required for oils having viscosities at 100° F. of 100/200 secs. and 200/500 secs. are 40/50 and 50/60 respectively, whilst for bright stocks and cylinder stocks 70% is required. For the corresponding solutions, quantities of 20, 30 and 40 lb. of filter aid per barrel are suggested as starting points.

The filter aid must not contain more than 0·5% of water, or plugging of the filter leaf will occur on account of ice formation, etc. The oil/naphtha solution is cooled to a temperature of 20° F. below the desired pour point and the filter aid is then added and mixed until a smooth, lump-free slurry is obtained.

The filter leaf is cooled in naphtha to a temperature similar to that of the mixture and then transferred to the solution tank. Filtration is carried out under vacuum and the period is timed (2 min. is recommended). The filter leaf is then removed from the tank, and the oil solution in the cake removed firstly by application of vacuum, and secondly by drawing chilled naphtha through the leaf and again applying vacuum. These portions are kept separate from the original filtrate as their pour points are liable to be higher.

The effects of the variables (*a*) percentage naphtha dilution, (*b*) rate of cooling the oil/naphtha solution, especially between the temperature range of 10° F. above the cloud point of the oil and 0° F., (*c*) quantity of filter aid added, on (1) the yield of oil of required pour point, (2) the rate of filtration and (3) the loss of lubricating stock, should be investigated.

From a consideration of the data obtained the conditions necessary for the most efficient application of the process can be ascertained. R. A. E.

367. The Influence of Different Fats on the Viscosities of Compounded Oils Compared with those of the Mineral Oil Components. E. Pyhälä. *Petr. Zeit.*, 1931, **27** (3), *Motorenbetrieb* (1), 3–5.

A number of compounded oils of American origin were utilised in this research and various animal and plant fats or oils, bone fat, lard, cotton-seed oil, blown rape-seed oil, etc., as fatty components. The types and percentages of fats added, the sp. gr., f. pt., viscosity in ° E. of the compounded lubricants and of the mineral oil components are given. The viscosities of the compounded lubricants are compared with those of the mineral lubricant base at corresponding temperatures.

The results show that the addition of fats which are solid at ordinary temperatures, *e.g.*, lard and bone fat, and which have fairly high viscosities (4·4–5·2° E.) at 50° C. cause a decrease in the viscosity in the compounded mixture at 100° C. The addition of animal fats to cotton-seed oil produces an increase in viscosity of the compounded mixture but not such a marked increase as the addition of a very viscous rape-seed oil to a mineral lubricant. W. S. E. C.

368. Action of Treating on the Oxidisability and Oiliness of Lubricating Oils. N. I. Chernozhukov and A. M. Gutzait. *Neft. Khoz.*, 1930, **18**, 806–817.

Emba spindle oil had the highest oiliness when treated with 15% of fuller's earth. The degree of wetting is independent of the acidity of the oil. The colour of the oil is directly proportional to the amount of fuller's earth used.

A treatment with 5% of silica gel is equivalent to a treatment with 18% of fuller's earth. Oxidation tests carried out by blowing oxygen at 120° through the oil showed that Emba distillate forms a precipitate of an asphaltic character after being treated with fuller's earth at 50°, while Baku spindle-oil distillate separates hydroxy acids under the above conditions. A fuller's earth treatment together with air agitation at 150° is more efficient and absorbs the asphalt more rapidly. Both oils on treatment with 3% of sulphuric acid produce a stable product provided that the suspended matter has sufficient time to settle out. An additional treatment with 10% alcohol solution improves the stability. A stable oil is then obtained after fuller's earth treatment. A quite stable Emba spindle-oil distillate is obtained after treatment with 1% of acid and 2% of silica gel, while for Baku distillate 1% of acid and 2–3% of fuller's earth are required. The addition of 1% of sulphonic acids to Baku machine oil makes it resistent against oxidation. Eighty per cent. of fuller's earth is required to stabilise Baku distillate if this is the only treatment. Residual fuel oil (mazout) was treated with 10% of sulphuric acid at 60° and the upper layer taken out and treated with 10–15% of fuller's earth at 200° for half an hour. A stable oil was obtained.

W. S. E. C.

369. Voltolisation as a Factor in Improving the Oiliness of Lubricants. F C. Otto. *Petr. Eng.*, **2** (4), 112–117.

Interest in the "Voltol" process for producing lubricants is anticipated. The practice of recent years has been to prepare base products which are added to mineral oils in percentages varying from 1/25, according to the specification desired. The two base products are a voltolised mineral oil of maximum viscosity 70° E. at 50° C. and a voltolised mixture consisting of 90% mineral oil and 10% fatty oil, having a maximum viscosity of 90° E. at 50° C. A description of the method of preparation of the base products is given. In addition, high grade transformer oil has been prepared for a number of years in a European plant by applying the "Voltol" process to Russian and New Jersey water-white kerosines of 43/46 gravity.

Modern engines in many cases demand an oil of higher viscosity and improved "oiliness" as compared with older models, and the "Voltol" process provides one method of obtaining improved "oiliness." Furthermore mixtures of mineral oil and voltolised oil possess a very flat viscosity curve and this property is useful for compounding airplane, tractor and motor oils. Another advantage possessed by such blends is that an oil giving satisfactory lubrication in hot weather will be sufficiently fluid to serve in cold weather.

Reports on tests carried out in various types of engines, turbines and electric generators, show savings in power and in lubricating oil costs and consumption and a reduction in frictional heat of 25/55%.

As a compounding agent for steam cylinder oils, votolised oil compares favourably with tallow and lard in regard to emulsifying power and has the advantage that blends do not tend to become rancid or to oxidise easily. References to literature on voltolising are appended.

R. A. E.

370. Observations on the Testing of Lubricants. E. Wooler. *J. Soc. Aut. Eng.*, 1931, **26** (1), 53.

The pressures to which automobile rear-axle gear teeth are subjected have increased 50% or more for some cars since 1924. Loads of 1800 lb./inch of tooth length are now not uncommon. The Timken Roller Bearing Co. has built two machines for testing gear lubricants, the one to determine scuffing or scratching, the other abrasion or general wear. In the machine to deter-

mine the load-carrying capacity of a lubricating oil before scuffing begins is a mandrel, at one end of which a Timken bearing cup is held firmly in place by a nut. Below the cup is a test block supported by a lever which is itself supported on a knife edge by a second lever. The first lever is loaded with weights to apply any desired pressure at the test block. The second lever indicates the coefficient of friction when it is balanced by weights. The oil flows over the test block by gravity and is pumped back to the elevated reservoir. The rubbing speed is 320 ft./min., and a test is usually run for 30 min.

With no sulphur and a low content of lead soap, the load-carrying capacity is very low. Lead-soap and sulphur content should vary inversely to give constant results; but a high content of lead soap is very difficult to hold in permanent suspension. Free sulphur in an oil confers high load-carrying capacity but leads to abrasion and tends to separate from the oil. Castor oil has no more load-carrying power than straight mineral oil. Changing the rubbing metals had little effect on the load at which scuffing began.

The machine for determining wear or abrasion showed a total wear of 0·0015 in. on the pinion shaft bearings in 200 hours running with a straight mineral oil. A Smackover oil containing 3% of combined sulphur produced little more abrasion than mineral oil without sulphur. Lead soap and copper oleate oils caused wear and separated badly. W. S. E. C.

371. Study on the Oxidation Phenomena of Insulating Mineral Oils. I. S. Mizushima and T. Yamada. *Res. Electro. Tech. Lab., Tokyo*, No. 291, 1930, 18 pp.

A slight portion of sulphur amounting to 0·1% was added to a refined white transformer oil and the oil was then tested for oxidation during 100 hours under the pressure of 86 cm. of mercury at 120°. The test of the oil without sulphur was carried out parallel with this test. The oil without sulphur oxidised readily, but the one with sulphur added did not oxidise appreciably. Alkyl sulphides and mercaptans, when added to the oil, give the same anti-oxidation effect, while thiophene does not. Therefore the authors consider that the stability toward oxygen of oils that are not highly refined must be attributed to the influence of sulphur compounds contained in them. It is, however, another problem how the additional sulphur influences the oils which are not highly refined and which produce sludge when oxidised. There are two conflicting opinions with respect to the effect of sulphur upon the sludge-forming character of the oil, namely, that it is acceleration, and that it is retardation. The authors' experiments made with seven different kinds of oil show that in all cases the oil with 0·2% of sulphur added formed more sludge than the oil without sulphur added. Transformer oils that produce a large amount of sludge always contain certain amounts of sulphur and unsaturated compounds. The authors also investigated the rôle of these sulphur and unsaturated compounds in the oil in the formation of sludge. To obtain the oil with some unsaturated compounds but free from sulphur, highly refined transformer oils were submitted to cracking at high temperatures. With the oils thus treated oxidation tests were carried out with and without addition of sulphur. In the cracked oil without sulphur sludge did not form, whereas acidity formation and other phenomena which prove that oxidation has taken place remained nearly the same as in the uncracked oil; the cracked oil to which sulphur was added gradually darkened in colour, though its acidity did not increase, and finally the oil precipitated a remarkable quantity of sludge. The authors consider, therefore, that both the sulphur and the unsaturated compounds are essential for the formation of sludge in the oil. W. S. E. C.

372. Correlation of Specifications for Transformer and Switch Oils. Typke. *Erdol und Teer*, 1931, 7, 42–44 and 59–60.

The specifications existing in various countries for transformer and switch oils constitute a serious obstacle to trade. The greatest differences are in resistance to deterioration tests and the International Electrotechnical Committee has taken steps towards securing uniformity. Oils from different sources and refineries have been sent to a number of countries and there investigated with regard to tests and resistance to change in transformers. It is to be hoped that this investigation will clear the way for further work.

No steps appear to have been taken hitherto towards unifying the other points of the specifications, although much less difficulty exists here.

This paper discusses how far it is possible to unify these points in the specifications current in various European countries, together with those of the Bureau of Standards in the U.S.A. The details of the specifications are then given, with the exception of the tests for homogeneity, and the various points are then discussed separately.

With regard to sp. gr., England, Italy, Russia, Switzerland and the U.S.A. make no mention; in other countries specify gravities from 0·85 to 0·92 at 20° C. Any ice which may be formed will only sink in oils with gravities below these limits. Ice rising or floating on the surface may cause flashing. In most countries 0·92 gives a sufficient difference in gravity to facilitate the rapid settling out of water. The lower gravities are favoured for open-air transformers. It would thus seem desirable to fix a limit of 0·92 at 20° C. in all countries except for countries with periods of great cold, and for open-air transformers and switches, when 0·895 is proposed.

Approximate conversions are given from Engler, Saybolt and Redwood viscosities at various temperatures to absolute units at 20° C. It is widely thought that thinner oils are better cooling media, but experiments by the A.E.G. have shown that there is no appreciable difference at working temperatures. A viscosity of 6° E. (0·451 absolute) maximum is considered sufficient.

For transformer oils the open fl. pt. is roughly 10° higher than the closed values. An open-flash of 145° C. or 135° Penaky is considered sufficient. An oil with this fl. pt. is as safe as one with 300° fl. pt., against inflammation from other than electrical causes and, if arcing occurs, there is no greater resistance with the high-flash oil than with the lower.

The method of test is very important when specifying the solidification point, and the figures given are not directly comparable. A description of the methods employed in various countries is given. No general uniformity can be expected here as some countries have long periods of frost to contend with. For these countries a solidification point of —40° C. in a 15 mm. tube for outdoor switches and —15° for other purposes together with a viscosity under 50° E. (3·4 absolute) at —5° C. would meet all just requirements.

Acid numbers are given in various ways which may be related: 1% SO_3=1·2% H_2SO_4=7% oleic acid=14 mgm. KOH per gram. A maximum of 0·05 mg. KOH per gram is recommended.

Some countries allow a maximum of 0·01% ash, but it is considered that the oil should be entirely free from ash. No specification for loss by volatilisation is thought to be necessary, as the flash-point stipulation is considered to provide for this point. Tests for sulphur and saponification values are considered unnecessary.

There are no marked differences in details of construction of transformers and switches in different countries which would make the adoption of a uniform grade of oil impossible, nor would it result in limiting the supply of oils available for this purpose and thus cause a rise in prices. E. B. E.

373. Lead Base Lubricants. J. A. Edwards. *J. Soc. Aut. Eng.*, 1931, 27 (1), 31.

Gear lubricants are required for automobile transmissions and axles that will form a film capable of withstanding the heavy pressures to which the gear teeth are now subjected. Lead base lubricants (containing lead soaps) have sufficient oiliness to enable them to form such films. Sulphur is also added to assist in protecting the gear teeth. The following is the mean of three manufacturers' specifications: sp. gr., 1·0; fl. pt. (min.), 320° F.; fire point (min.), 360° F.; viscosity (Say.) at 211° F., 90–105; pour point, 15° F.; lead soap (min), 12%; free lead, none; free oleic acid, 0·5%; sulphur, 1% (max.); and added sulphur, 2·5%. Machines for breakdown tests have been developed, such tests being made under pressures up to 20,000 lb./sq. in. The breakdown point is taken as that at which abrasion of the bearing surface occurs. The coefficient of friction and the rise in temperature are also determined. Suggested substitutes for the lead soap are aluminium stearate and sulphurised oils. W. H. T.

374. Crystallisation of Petroleum Wax. F. W. Padgett. *Petr. Eng.*, 1931, 2 (4), 80–84.

References to the literature on the subject of the crystallisation of petroleum waxes and a brief summary of the theories and conclusions arrived at by the various investigators are given. The following conclusions are drawn by the author: (1) The microscope gives valuable information concerning the suitability of products for industrial operations, particularly sweating and pressing. (2) Crystallisation is affected by cooling rate, viscosity of medium and especially by the presence of impurities which modify or inhibit crystallisation. The impurities are adsorbed or occluded in the crystallising wax, thus affecting the crystal habit. (3) Rate of cooling has a greater effect on crystal size in the lower than in the higher distillates and residues. (4) The wax crystals are in the orthorhombic system. (5) Marked branching of the chains may cause the hydrocarbons to become soft or amorphous and the surface tension overcome the tendency to form crystals. (6) Probably waxes in the lower lubricating oil distillates are mainly straight chain hydrocarbons. (7) The mode of crystallisation is of such nature at times that oil could readily be enclosed in the wax. (8) The crystal habit of the waxes is as follows: plates, needles and a fibrous feathery form. Plates are the most stable form. (9) Finely divided and colloidal substances have a definite effect on the crystallisation. (10) It is possible that the higher the molecular weight of the wax the less definite is the tendency to form good crystals. (11) In "amorphous" wax is it not impossible to have soft wax crystals and heavy oils, also a smaller proportion of hard wax finely dispersed. It is not necessary to assume that crystalline wax is produced by cracking amorphous wax; thus in preparing lubricating oil distillates for pressing, carefully controlled fractionation without cracking is now employed. (12) Dilution favours plate crystals; rapid cooling favours small crystals and needles; high viscosity of medium favours smaller crystals and needles; impurities favour needles, fibres and "amorphous" wax. R. A. E.

375. Non-freezing Lubricants. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 340,249, 27.7.29.

Non-freezing lubricants can be prepared by mixing oils and fats with polyhydric alcohols or esters, or ethers of such alcohols with themselves or other alcohols; for example, ethylene glycol monoethyl ether, di-ethylene

glycol or similar polyglycols, dioxane, etc., in presence of dispersive agents, such as Turkey red oil, alkylated naphthalene sulphonic acids, etc. A typical recipe is: 120 kg. acids of tallow, 80 kg. olein, 400 kg. mineral oil, 80 kg. ethylene glycol monoethyl ether, 39 kg. KOH and 100 kg. water. R. W. L. C.

376. Preparation of High-molecular Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 340,293, 17.7.29.

Hydrocarbon oils of high molecular weight (lubricating oils) are produced by polymerising lower molecular unsaturated hydrocarbons at temperatures lower than those at which splitting of the initial or resulting product might occur. The reaction is carried out in the presence of complex acids which contain boric acid as one constituent and as the other, an organic substance containing two or more hydroxyl, carbonyl, or carboxyl groups on the same or on two adjacent carbon atoms. The process is carried out at super-atmospheric pressures, the initial material being diluted with saturated hydrocarbons. The complex acid is added to the extent of from 1–30% by weight of the initial material. W. H. T.

377. Apparatus for Sweating Wax. H. L. Allan. E.P. 340,993, 2.10.29.

The apparatus consists of a vessel provided with means for charging with wax and for supercharging additional wax to compensate for the shrinkage which occurs during cooling. The wax is supported within the vessel without the use of water as a wax support. The wax container is a deep closed vessel externally insulated so that the operation does not require a specially heated building, and is provided with a large number of transverse or horizontal pipes arranged in rows. The pipes in each row are staggered in relation to those of the rows above and below. Heating and cooling fluids may be circulated through the pipes from inlet and outlet chambers connected at their ends and common to a group of superposed rows of pipes. The method of arranging the pipes provides an efficient support for the mass of wax and leads to improved heat transference. The bottom of the vessel may be dished and corrugated and a false bottom of reticulated metal or metallic gauze is inserted to form a support for the mass of wax. An outlet is provided at the bottom for discharging the oil and liquefied wax. The plant may also be in the form of a closed upright vessel divided by partitions into superposed compartments with a wax support above each partition and straight transverse heating and cooling pipes in staggered rows. R. W. L. C.

378. Filtering Suspended Sediments (Sludge). C. Pontoppidan. E.P. 341,176, 11.11.29.

The filter consists of a perforated pipe covered with filter cloth so arranged that it can be withdrawn from and pulled into a pressure tank by means of a piston rod. The sludge to be filtered is fed into the pressure tank so that the solid builds a cake around the filter body. When a suitable cake has been deposited, the filter is withdrawn from the pressure tank through an opening, the wall or edge of which fits like a stuffing box around the filter body and the cake deposited thereon. Unfiltered sludge is removed by the edge of the stuffing box and the filtered sludge is removed by a scraper. The filter body with the deposited cake may be drawn into a chamber in which washing or drying of the filter cake can take place. The plant may be adapted for continuous filtration by constructing the filter body as an endless flexible member, which is supported and driven by drums. The pressure tank is then fitted with packing devices at both ends. R. W. L. C.

379. Separation of Fatty or Wax-like Bodies from their Solutions. J. K. Pfaff, K. Böttger and A. Sieweke. U.S.P. 1,779,287, 21.10.30. Appl., 2.7.29.

Soft wax (petroleum jelly), paraffin wax and Montan wax is separated from solution in petroleum distillates, Montan wax distillates and similar distillates, and organic solvents, by dissolving in the solutions, while hot, an amount by weight approximately equal to that of the wax-like bodies to be separated of a substance which crystallises well, and is soluble in the same solvent, but differs chemically from the bodies being separated. The resulting solution is cooled to at least 0° C., whereby simultaneous precipitation of the wax-like bodies and the added substance is effected. The solvent is removed by filtration, and the precipitated substances separated into the added substance and the required wax-like body by distillation. The substance added during this process to the solution containing the wax-like body is preferably naphthalene. H. E. T.

380. Fractional Extraction of Petroleum Hydrocarbons with Alcohol. T. A. Werkenthin. U.S.P. 1,783,203, 2.12.30. Appl., 30.12.27.

Heavy petroleum oils, wax-bearing distillates and hydrocarbons are treated by successively extracting with alcohol at temperatures of from 100° F. to the b. pt. of the alcohol in a mixing coil by passing preheated oil and alcohol through the coil. The hot mixture passes first to a settling tank and then to a centrifugal separator from which the separated alcoholic solution proceeds to a cooling coil and from thence to separating tank. The solution there deposits the extracted oil precipitated by the drop in temperature. The alcohol is returned to circulation. It is claimed that this method of recovering the dissolved oil by cooling the solution instead of by distillation, as previously suggested, is a more economical process and results in the production of a better-coloured extract. R. W. L. C.

381. Dewaxing Petroleum Oil. L. D. Jones. U.S.P. 1,784,525, 9.12.30. Appl., 7.10.25.

The steps in the process consist in the production of a long residuum of petroleum containing the lubricating oil fractions, the reduction of the hard asphalt content of refining with acid or clay or by distillation, the dilution of the oil with naphtha followed by chilling and centrifuging for the removal of wax and subsequent distillation and finishing for the production of lubricating oils.

The particular feature of this invention relates to the preparation of the stock for dewaxing so that satisfactory centrifugal separation of the wax will occur. Objectionable or hard asphalt must be removed from the oil for, if present, it will precipitate out on dilution and chilling and affect both the precipitation of the wax and its subsequent discharge from the centrifuge bowl. The purification of the oil by acid or by clay treatment is regulated so that sufficient of the normal impurities present in the petroleum are retained to have an "amorphizing" effect on the precipitated wax. Oil containing crystalline wax and deficient in the impurities which favour the precipitation of wax in the amorphous form may be blended with oil containing an excess of such impurities in order that the wax precipitated during dilution and chilling may be in a sufficiently plastic form to permit centrifugal separation to be satisfactorily applied. R. W. L. C.

382. Preparing Petrolatum. F. Lavirotte. U.S.P. 1,785,270, 16.12.30. Appl., 30.9.25. Conv. (Fr.) 4.10.24.

Substantially pure and white stable petrolatum is made by treating crude petrolatum with a strong H_2SO_4 at a temperature of about 70° C. for about

24 hours, increasing the temperature to about 160°–170° C. for about 48 hours to carbonise the sulphonated compounds. The liquid material is subjected to a second acid treatment whereby residual sulphonated compounds are carbonised. Carbonaceous material is then removed and the liquid purified by filtering through finely divided clay. R. W. L. C.

383. Regeneration of Lubricating Oils. H. Bensmann. U.S.P. 1,787,353, 30.12.30. Appl., 2.11.26.

Used lubricating oils are first heated to 30° C. and then gradually raised in temperature to about 60–70° C., while adding polymerising agents such as sulphuric acid. Asphalt-like substances are formed from the decomposition products of the oil and are drawn off as sludge. The treated oil is passed through a settling vessel and then filtered through an aluminium magnesium hydrosilicate to remove the organic acids and tarry constituents.
W. H. T.

384. Refining Lubricating Oils. J. C. Black and W. D. Rial, Assrs. to Richfield Oil Company. U.S.P. 1,787,505, 6.1.31. Appl., 27.4.27.

Lubricating oil stocks are heated at a temperature of approximately 280° F. with 5% of 10° Bé. caustic potash solution under a pressure of approximately 50 lb. gauge. The mixture is allowed to separate and the oil layer washed with water until practically free from potash. The oil is then refined with sulphuric acid and neutralised by contact filtration with adsorbent clay and subjected to vacuum distillation whereby the higher grades of lub. oil are separated from the cylinder stock. The process is particularly suitable for refining the products from asphalt base petroleum oils.
R. W. L. C.

385. Bleaching Mineral Oils and Fats. W. Posth and F. Besemann, Assrs. to I. G. Farbenind. A.-G. U.S.P. 1,788,204, 6.1.31. Appl., 19.8.27.

Mineral oils are subjected to the usual acid and alkali refining treatment and then treated with an aqueous alkaline hypochlorite solution containing a bicarbonate. The alkalinity of the hypochlorite solution is reduced by the bicarbonate and decomposition of the hypochlorites into chlorides and oxygen is accelerated by the addition of a salt of the metals copper, nickel and cobalt. The mixture of oil and the activated hypochlorite solution is heated to about 60° C. The hypochlorite solution, of which 4 to 5% by weight is used, should contain about 150 gr. of available chlorine per litre. The oil is separated from the spent bleaching liquor and washed. R. W. L. C.

See also Abstracts Nos. 350, 405.

Fluxes and Asphalts.

386. Make Asphalt by Vacuum Reduction. D. H. Bancroft. *Oil and Gas J.*, 1931, **29** (36), 30.

The Lion Oil Refining Co. are making asphalts by vacuum reduction from heavy Smackover Crude in their El Dorado (Ark.) refinery. The plant consists of a new 6,000-brl. combination dehydrator topping unit and vacuum reduction tower, the products made being gasoline, gas oil, lub. oil distillate and asphalt.

The crude, which contains a considerable amount of water in suspension, is first passed through a furnace to the dehydrator flash tower, maintained

under a partial vacuum, where the water and a small volume of light gasoline is removed. The dehydrated crude passes through a vapour and residue heat exchanger and it is then heated to 680° F. before being pumped to a second flash evaporator. The tops from this go to a bubble tower, where the remaining gasoline is removed, gas oil being taken from the bottom. The residue passes through a second series of coils in the furnace where its temperature is raised from 665° to 765° F. The oil is then taken direct to the vacuum tower, where it is flashed under 27½ mm. abs. pressure. The tower is 17 ft. inside diam. and 44 ft. 9in. high, with a superstructure 25ft. above this to accommodate the exchanger, condenser and ejector. From the top of the tower a lub. oil distillate is taken, asphalt being removed from the bottom.

The plant has also been used to run Smackover residuum containing 10 to 15% b. s. & w. mixed with an equal proportion of Smackover crude to produce various standard grades of asphalt.

Particular attention is being paid to the preparation of cut back asphalts for cold patching and cementing roofing materials, and for jointing paving. A flow sheet and general view of the plant is given. C. L. G.

387. Separation of Bitumen from Bituminous Sands. K. A. Clark. *Nature*, 1931, **127**, 199.

The Research Council of Alberta has found that good separation can generally be affected by mixing the sand with about one-fifth of its weight of a 2% solution of sodium silicate—heating to 85° C. and then adding this mixture to water at the same temperature.

The bitumen collects on the surface.

Sand containing 10-17% of bitumen gave a product containing less than 5% of mineral matter.

Some difficulty was experienced in duplicating the results and investigation showed that this was due to acidity picked up from the sand by the water. (Probably the acidity is derived from ferrous and ferric salts.)

Adjusting the *p*H value of the water to 6·4 by means of sodium hydroxide or sodium silicate gave much better results. A preliminary wash of the bituminous sand with cold water diminished the quantity of alkali necessary, and removed any clay.

The investigation is being continued. C. C.

See also Abstract No. 448.

Special Products.

388. Production of Liquid Hydrocarbons from Propylene. A. Mailke and Renaudie. *Compt. rend.*, 1930, **191**, 851–854. (*Br. Chem. Abs.*, 1931, p. 60.)

The deposition of carbon during the reaction of ethylene with silica gel at 700° C. is due to the presence of metallic iron. When this is removed, the liquid products consisting of ethylenic hydrocarbons, benzene and toluene, are obtained in high yields, methane and its homologues being produced only in small quantities. Naphthalene and anthracene have been isolated from the tar. Low-boiling olefines, aromatic hydrocarbons and a tar in which toluene, *m*-xylene, naphthalene and anthracene have been detected, are formed when propylene is passed over silica gel at 650° C. under similar conditions. W. H. T.

389. Determination of Ethylene Oxide in Gas Mixtures. W. Deckert. *Z. anal. Chem.* 1930, **82**, 297–307.

Mixtures of ethylene oxide (A) and carbon dioxide are used under the name of T-gas for killing insects. When A reacts with hydrochloric acid glycol chlorohydrin is formed, one molecule of A reacting with one molecule of hydrochloric acid. The reaction can be made the basis of a volumetric method for determining A in a gas mixture. By passing the gas through a 22% solution of sodium chloride in 0·1 *N* hydrochloric acid all of A is absorbed and excellent results are obtained by titrating the excess of HCl if the solution is first heated; or the diminution in volume of the gas after treatment with HCl—NaCl solution can be used as a basis for the analysis. For a practical analysis of air that has been sprayed with A, a slow stream of the air is passed (1 litre in 4 min.), through 0·1—0·001 NHCl until at least 0·44 mgms. of A have been absorbed. The solution is then heated to about 70° and the excess acid titrated with caustic soda solution, using methyl orange as an indicator. 1 c.c. of 0·1 *N* HCl≡4·4 mgms. of A. W. S. E. C.

390. Solvents. T. H. Durrans. *Oil and Colour Trades J.*, 1931, **79**, 177–182.

Having referred to "low," "medium" and "high" boilers and the effects of azeotropes formed when a number of miscible components are employed in cellulose lacquers, it is stated that solvents miscible with water (acetone, etc.) may be used according to circumstances. Water added to such solvents (for the sake of cheapness) should be regarded as a non-solvent diluent such as benzene, toluene or the paraffins. In any lacquer, in which the amount of diluent approaches the maximum permissible, then the addition of more diluent (hydrocarbon, alcohol or water) in sufficient quantity will cause precipitation of the solutes. It is preferable, in any case, for the quantity of diluent to be so limited that room is left for more in the form of water deposited from the atmosphere.

The qualities of the various classes of solvents are (*a*) low-boilers of high solvent power to enable preparation of concentrated solutions of low viscosity and to obtain quick initial drying; (*b*) medium-boilers to check evaporation rate and thus to reduce defects caused by excessive rate of evaporation and also to impart good flowing properties; (*c*) high-boilers to protract period of drying for long periods and thus to impart good brushing properties and gloss; (*d*) plasticiser to knit all the constituents into a homogeneous whole; to impart flexibility, permanence, gloss and to reduce brittleness; (*e*) diluents to reduce cost and control viscosity. C. I. K.

391. Catalytic Production of Esters. Du Pont de Nemours and Co. E.P. 313,575, 14.6.29.

Esters are produced by the catalytic dehydrogenation of alcohols, a process which also yields considerable quantities of the corresponding aldehydes. Aldehyde formation may be reduced to a minimum by using suitable catalysts, such as dehydrogenating oxides, *i.e.*, oxides which preferentially dehydrogenate alcohols to yield aldehydes rather than olefines, *e.g.*, zinc oxide, manganese oxide, magnesium oxides or mixtures of these. These oxides may be mixed with other oxides less active but more acidic than zinc oxide, *e.g.*, the oxides of vanadium, chromium, tungsten, molybdenum or uranium. Alternatively, the latter series of oxides may be displaced by an easily-reducible oxide in such amounts and under such conditions that the metals are maintained substantially in oxide form throughout the process, although in the presence of reducing gases. Examples of the process are given, such as the production of ethyl acetate by catalytic dehydrogenation of ethyl alcohol under a pressure of between 70 and 280 atm., at a temperature of from 385° C. to 425° C., and a space velocity of 16 vol. of alcohol per vol. of catalyst per hour. W. D. S.

392. Polymerisation of Unsaturated Organic Compounds. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 340,004, 12.8.29.

The polymerisation of unsaturated organic compounds can be accelerated by carrying it out in the presence of metal carbonyls. The carbonyls of iron, nickel and cobalt are particularly suitable but those of molybdenum, tungsten or chromium may also be used. Unsaturated fatty acids and unsaturated hydrocarbons with more than one double linkage may be polymerised in a very much shorter period of time by this method than by the employment of high temperatures and pressures. The quantity of carbonyl employed varies from 0·1 to 10% with the nature of the material and of the carbonyl employed. Cobalt carbonyl is particularly efficient. The initial materials employed need not be monomers or dimers but may be products which have already attained a certain degree of polymerisation. Solvents may be advantageously used in the polymerisation of highly viscous products. Addition of oxygen, or agents supplying oxygen, such as the heavy metal salts of sulphonic acids of organic compounds, is preferable when polymerising partially polymerised diolefines. W. D. S.

392. Polymerisation of Diolefines. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 340,008, 19.8.29.

The polymerisation of diolefines in the presence of metallic polymerising agents is improved by employing additions of organic compounds in which at least two valencies of the same carbon atom are saturated by oxygen. Examples of such compounds are acetals, in particular cyclic acetals obtained by condensation of 1·2- or 1·3-glycols with aldehydes, ketones such as acetone, cyclohexane, quinone, furfurol, and aldehydes, especially unsaturated aldehydes. Esters and carboxylic acids also have a beneficial effect. As a general rule, small amounts, between 5 and 0·05% by weight of diolefine, of the organic compounds are added and the metallic polymerising agent should always be in excess. The process is operated between room temperature and 90° C. By suitable selection of conditions and of the proportions of the selected organic compound, it is possible to prepare products suitable for many uses. W. D. S.

394. Absorption of Olefines in Sulphuric Acid. Standard Oil Develop. Co. E.P. 340,098, 14.11.29.

Reactive acid liquors are produced from mixtures of olefines of different mol. wt. by treating the olefines with H_2SO_4 in three successive stages. The acid for the first stage is below 65% strength and the temperature is regulated between the limits of 90° and 120° F. for the absorption of isobutylene and its higher homologues. In the second stage, secondary butylene, amylene, hexylene, etc., are absorbed in an acid of between 78 and 82% strength, preferably 80%, at a similar temperature. After removal of the higher olefines, propylene is absorbed in the last stage with concentrated acid of above 90% strength, preferably 93%. The temperature should be about atmospheric. A modification is suggested in which the entire operation takes place in one stage, the requisite amount of water being added to the acid at certain stages as it descends through a tower. The olefine—containing gas ascends through bell cap plates at such a rate that the several olefines are completely absorbed by the various dilutions of acid. Cooling or heating is arranged by means of coils. When gaseous mixtures of olefines, it is preferable to add a mineral oil in the proportion of 3 vol. per vol. of acid, to the top of the tower. By this method the formation of polymerised olefines is diminished and the quantity of acid used reduced. Olefines higher than propylene may be absorbed

up to about 1 mol. of olefines per mol. of acid. After hydrolysis as much as 150 to 170 vol. of higher secondary alcohols per 100 vol. of 80% H_2SO_4 have been obtained and even greater yields in case of tertiary alcohols.

W. D. S.

395. Production of Carbon Black. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 340,239, 17.9.29.

Gaseous carbon compounds are subjected to decomposition in a rotary vessel preferably provided with perforated walls for leading off the carbon black formed. A catalyst is present in a freely moving state, having the shape of rotation bodies. W. H. T.

396. Storing and Transporting Volatile Pesticides. Heendt-Lengler G.m.b.H. and J. Lengler. E.P. 340,343, 8.8.29.

Volatile poisonous pesticide is transported in a metal vessel containing porous material on which it is absorbed. Air is withdrawn from the vessel and replaced either by inert gases or by vapours of the pesticide. A. R. S.

397. Producing Carbon Black from Oil Refinery Residues. W. W. Trigg. E.P. 340,482, 19.12.30. Appl., 19.9.29.

The heavy residues remaining after cracking of petroleum oils are cracked at high temperatures and under low pressure. The resulting highly unsaturated gases are added to the gas formed during the usual high pressure cracking operations. The mixed gases are burned with incomplete combustion to produce carbon black. W. S. E. C.

398. Manufacture of Styrenes and their Homologues. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 340,587, 30.12.30.

Styrene and its homologues are produced by the dehydrogenation of aromatic hydrocarbons having an aliphatic side chain with at least two carbon atoms. The operation is carried out at a temperature between about 500° and 800° C. in the presence of a suitable dehydrogenating catalyst. The catalyst may comprise metal compounds which are difficultly reducible under the conditions of working, and if desired, a carrier. The addition of small amounts, such as from 1 to 3% by weight of the catalytic substance, of metal compounds which are reducible under the reaction conditions, *e.g.*, copper and iron oxides, frequently increases the activity of the catalysts. The catalysts have a longer working life if preheated in the presence of a gaseous compound of carbon or of another gaseous agent consisting of or containing hydrogen or of nitrogen. A modification of the process consists in employing a dehydrogenation catalyst such as a metallic oxide incompletely reducible under the conditions of working and preheating the catalyst in the presence of a gaseous reducing agent until the metal oxide is reduced to a lower stage of oxidation. Examples are given with yields of styrenes varying between 20 and 70%. W. D. S.

399. Synthesis of Alcohols. G. Natti. E.P. 340,656, 8.1.31.

Some difficulty has been experienced hitherto in finding satisfactory catalysts for the synthesis of methanol from water gas. It has been found that smithsonite, preheated to 400° to 500° C., is capable of absorbing large quantities of concentrated solutions of alkalis or alkaline salts of fatty acids. Such a catalyst is capable of yielding much increased yields of alcohols from carbon monoxide and hydrogen. The pressure employed is from 250 to 300 atm., the temperature about 400° C. and a gas velocity corresponding to 10 cm. per hour (measured at ordinary pressure) per litre of gross catalyst space.

Smithsonite alkalinised with 20% of potassium hydrate yields a mixture half composed of ethyl and propyl alcohols, not more than 20–25% of methanol and small amounts of higher alcohols. Sensible quantities of other organic bodies are not obtained and there is no indication of methane. The results obtained with smithsonite activated with potassium acetate are better than those obtained with potassium hydrate. A yield of alcohols almost 50% more than that obtained with alkali was observed but the mixture contained a greater quantity of higher alcohols. The gas mixture employed should contain more than 2 vol. hydrogen per vol. of carbon monoxide. Carbon dioxide which is detrimental to the formation of higher alcohols may be eliminated by a suitable washing or absorption apparatus.

W. D. S.

400. Manufacture of Insecticides and the Like of Low Water Solubility. Imperial Chemical Industries. E.P. 340,882, 18.3.30.

A process for the manufacture of manganese arsenate preparations for use as insecticides is described. Manganese dioxide in any suitable form and arsenious acid in an aqueous medium, are allowed to interact in a rotating autoclave or a pressure ball-mill, the surfaces in contact with the reaction mixture being of a non-ferrous nature, *e.g.*, of lead. The reaction takes place at temperatures above 100° C. under a steam pressure of about 80 lb./sq. in., and the product is treated subsequently under similar conditions, with hydrated lime in amount equal to 5–30% of the combined weights of the manganese dioxide and the arsenious acid. W. S. E. C.

401. Recovery of Oxygen-containing Derivatives of Unsaturated Hydrocarbons from Mixtures thereof with other Substances. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 340,984, 30.12.30.

Mixtures of unsaturateds with other substances are treated with complex compounds obtained from an anhydrous metallic or non-metallic halide which, when brought into contact with water, evolves heat, and an alcohol or organic acid. The treatment is conducted at such temperatures that additive compounds of the unsaturated organic substances only are formed with the complex compounds. The additive compounds are separated and decomposed, *e.g.*, with water

Using zinc chloride-acetic acid, alcohols of the formula $C_nH_{m-4}O$ were obtained, and with zinc chloride-ethyl alcohol, alcohols of the formula $C_nH_{2n-2}O$. W. D. S.

402. Oxidation Products from Hydrocarbons. C. Arnold and Standard Oil Develop. Co. E.P. 341,130, 15.1.31.

A gas rich in free oxygen is dissolved in the required hydrocarbon while in a liquid state and under pressure considerably over atmospheric. The mixture is passed through the reaction zone at a temperature between about 200° and 600° C. and the oxygen-containing derivatives are separated from the withdrawn products and unoxidised hydrocarbons returned to the process. In order to obtain the oxygen in solution the air and hydrocarbon in liquid state are passed in counter-current relation through a zone of contact while under pressure in excess of 100 lb./sq. in., preferably between 1000 and 3500 lb./sq. in., both during solution of oxygen and oxidation. The pressure and temperature should be so regulated in the contact zone that oxygen is dissolved in proportion less than $\frac{1}{2}$ mol./mol. of hydrocarbon. The mixture of hydrocarbon and oxygen may be preheated to a temperature not over 200° C. Liquid pentane yielded approximately equivalent amounts of acetaldehyde, acetic acid, ethanol, propanol, and smaller amounts of acetone and butanol. W. D. S.

403. Manufacture of Alcohols. H. D. Elkington and N. V. Bataafsche. Petr. Mij. E.P. 341,167, 15.1.31.

It is known that tertiary and secondary alcohols may be destroyed in part by the action of the acid in the reaction liquid obtained by the absorption of the corresponding olefine in sulphuric or other suitable strong acid. To overcome this drawback it has already been proposed to neutralise the acid reaction liquor with ammonia. It has now been found that the absorption of olefines in strong acid can be satisfactorily effected if the acid is diluted with an aqueous solution of the corresponding ammonium salt. After the absorption of the olefines the acid liquid is neutralised with aqueous or gaseous ammonia, or ammonium carbonate.

The extent to which the acid is diluted with the aqueous ammonium sulphate solution depends on the olefine to be absorbed. Examples are given for the preparation of tertiary butyl and amyl alcohols and it is shown that dilution of the acid before absorption has little effect on the yield of alcohol and gives much better absorption than if a corresponding amount of water had been used for dilution. The ammonium sulphate used is easily recoverable. W. D. S.

See also Abstract No. 448.

Refining and Refinery Plant.

404. Separation of Tars, Tar Oils, Petroleum and the Like into Fractions of Technical Value. J. Kárpáti. E.P. 336,945, 16.6.29.

Tars, tar oils, petroleum mixtures, etc., containing constituents of varying solubility are fractionated by bringing the raw material into a solution in a diluted aqueous-organic medium at an elevated temperature and by reducing the solvent capacity of the solvent in stages, whereby the dissolved substances are caused to separate into fractions of increasing solubility. The solution is affected so that pitch, bitumen, asphalt, etc., remains undissolved. The reduction capacity of the solvent is affected by reducing the pressure, the temperature or the concentration of the solvent, or by the addition of salts or by a combination of these methods. During the separation a temperature range of about 20°–150° C. is employed. The solvent consists of aqueous alcohol or aqueous alcohol-benzene, containing 30–70% of organic solvent. The fractions obtained are subjected to a treatment with a decolourising material. H. E. T.

405. Refining Petroleum and Like Hydrocarbons. W. W. Triggs, Assr., Pan American Petroleum Co. E.P. 338,483, 7.5.29.

The method applies to the refining of lamp oils, transformer oils, lub. oils, etc., derived from carbocyclic, asphaltic or mixed base crudes.

The raw distillate is first treated with liquid SO_2 at temperatures below 32° F. in a single contact tower. The temperature is preferably one at which the SO_2 has substantially no vapour pressure and is usually about 17° F. The partly purified oil is separated from the surplus SO_2, which contains the extracted undesired hydrocarbons, and is then treated with sulphuric acid or fuming sulphuric acid in quantities sufficient to complete the purification.

The acid treatment is carried out at temperatures below 60° F. and in the presence of the dissolved SO_2. It is claimed that a selective treatment is thus obtained without the extraction or chemical decomposition of any appreciable quantity of the desired refined product. The acid treated oil is settled from acid tar, freed from sulphur dioxide by vaporisation and then finished by a neutralising wash with alkali and filtration through fuller's earth, if desired. W. L.

406. Surface Apparatus for Heating or Cooling Liquids. R. Morton & Co. and P. Robinson. E.P. 339,869–70, 7.2.30.

These apply to heat exchangers having a tubular casing containing one or more inner pipes.

The inner fluid follows a sinuous path through the unit. This path is composed of a number of tubes connected together in series so that the fluid flows through the apparatus in a continuous stream. The tubes are mounted in tube blocks which are provided with passages or ports directing the flow of the outer fluid over the tubes. By confining the flow to a single stream through tubes of large diameter the flow is facilitated as the skin friction is considerably decreased.

The interior of the casing may also be sub-divided radially to provide a number of passages through which the outer fluid flows in succession.

W. L.

407. Finned or Gilled Tubes for Heat Exchangers. J. E. Gortner. E.P. 340,422, 23.1.30.

A heat exchanger or radiator unit consists of a tube of flattened section and gills arranged in pairs thereon. Each pair of gills is disposed on opposite sides of the tube and consists of a fin strip wound spirally on the tube and transversely slit from its outer edge at predetermined intervals. These intervals are substantially equal to the major diameter of the tube. The fin strip also has short longitudinal slits intersecting the transverse slits near the inner edge of the fin strip and the latter has a portion at its inner edge bent upon itself or rolled over a reinforcement. W. H. T.

408. Working-up the Waste Sulphuric Acid Produced in the Washing of Mineral and Tar Oils with Sulphuric Acid. Chemische Fabrik. Berk. G.m.b.H. and J. Grubenbecher. E.P. 340,816, 23.12.30.

Waste acid, without previous dilution, is led counter-current to a stream of tar oils in the vapour state. By this means a light liquid tar-oil resin mixture and sulphuric acid are obtained, and the concentrated acid is mixed with small amounts of water, the pure acid separating quantitatively from the tar-oil-bitumen mixture. W. S. E. C.

409. Tubular Heat Exchangers. W. Siller. E.P. 340,893, 31.3.30.

A high pressure heat exchanger is described in which a large number of juxtapositioned tubes are accommodated in a common jacket. An end wall is arranged in each end of the jacket through which the individual tubes extend and are tightly welded. The thickness of the end walls may differ from that of the walls of the welded tubes by not more than 2 mm., the same difference being allowed with regard to the distance between individual tubes and the wall thickness of the tubes. W. H. T.

410. Flue-gas Recirculation System for Shell Stills. H. A. Atwater. U.S.P. 1,779,405, 28.10.30. Appl., 16.2.28.

Each flue passage in a battery of stills is provided with a gate damper, and at some point along the flue gas tunnel connection is made for each battery, by a bend or elbow, with a suction fan for pulling a certain volume of the hot flue gases from the tunnel and discharging them into a header conduit. From here connection is made by branch conduits with a series of flue pipes, preferably two pairs for each still, which extend longitudinally through the still in such a position as not to interfere with the manholes and to afford reasonable access for cleaning. At the front end of each still return bends are connected to the ends of the flue pipes for reconducting the recirculated gases into the combustion chamber. W. L.

411. Purification of Hydrocarbon Oils. E. B. Hunn, Assr. to Standard Oil Development Co. U.S.P. 1,786,246, 23.12.30. Appl., 1.7.27.

Sour petroleum distillates are sweetened by treatment with doctor solution at a temperature above normal atmospheric temperature. Less than sufficient sulphur is added to completely precipitate the lead as lead sulphide and the aqueous spent doctor solution is withdrawn. A low boiling alkyl disulphide is then added to the distillate and the precipitated lead mercaptide is filtered. W. S. E. C.

412. Treating Hydrocarbon Oil. J. M. Goodwin, Assr. to the Texas Co. U.S.P. 1,786,650, 30.12.30. Appl., 16.3.28.

Cracked distillates are purified and stabilised by contacting with hydrogen sulphide until approximately 0·005 lbs. of the gas is dissolved per barrel of oil. The oil is then treated with sulphuric acid of a strength less than 66° Bé. The naphtha is freed substantially from hydrogen suphide before the addition of the measured amount of this gas. W. H. T.

413. Purification of Hydrocarbon Oils. C. P. Dubbs, Assr. to Universal Oil Products. U.S.P. 1,787,570, 6.1.31. Appl., 29.6.25.

Claims are made for a process of removing sulphurous compounds from hydrocarbon distillates in the presence of hyrdogen sulphide. The distillate is initially charged to a zone containing calcium hydrate and water wherein hydroxy hydrosulphide is formed as a reaction product. After passing through a second similar zone, the oil is returned to the first zone where the CS_2 content reacts with the hydroxy hydrosulphide formed in the first zone. A treatment with calcium hydrate and water serves to remove H_2S. W. H. T.

414. Treating Hydrocarbons. J. C. Black. U.S.P. 1,789,510, 20.1.31. Appl., 1.8.28.

The hydrocarbon oil is treated with liquid sulphur dioxide at low temperatures produced by an ammonia cooling system, and the treated oil is separated from the SO_2 extract. The separated products are subjected in separate containers to distillation operations at temperatures above 212° F. and under a pressure of about 100 lb. gauge. The major portion of the sulphur dioxide contained by the products is separated, condensed and collected. The remainder of the sulphur dioxide contained by the products is separated, condensed and absorbed in a hydrocarbon oil to be subsequently treated with liquid SO_2 by a second distillation operation in separate containers at pressures not greater than 10 lb. gauge and at a temperature above 212° F. W. S. E. C.

415. Refining Mineral Oils. A. Lachman, Assr. to Richfield Oil Co. U.S.P. 1,790,622, 27.1.31. Appl., 22.6.27.

This invention is particularly applicable to the refining of oils obtained by cracking processes. The oil is heated to approximately 150° C., the vapour being passed through an aqueous solution of zinc chloride maintained at a similar temperature, and having a concentration of approximately 70–85%. The vapours are subsequently condensed. The zinc chloride solution may contain 3% by weight of zinc hydroxide or 1–3% by weight of a mixture of zinc hydroxide and zinc oxide. W. H. T.

Cracking.

416. Separation of Sludge from Petroleum Residues. N.V. de Bataafsche Petr. Mij. E.P. 340,314, 26.9.29.

Residues, particularly from cracking operations, are mixed with diluents consisting of pressure distillates or pressure distillate bottoms, etc., in the proportion of 100 parts of residue to 50–300 parts of diluent. The mixture is maintained at elevated temperatures not exceeding 150° C. (90–100° C.), thereafter separating the sludge from the residue. W. H. T.

417. Apparatus for Treating Hydrocarbon Oils. W. W. Triggs for Panhandle Refining Co. E.P. 340,949, 29.6.29.

The plant consists of a cracking tube still and a preheater tube still, each in a separate chamber but contained in one structural unit. The furnace gases after passing upwards through the cracking coils enter the top of the second or preheating chamber. Adjustable dampers to the stack are provided above and below the preheater coils, so that more or less heat as required may be put into the oil. The cracking furnace is heated by Dutch oven type combustion chambers at either end, and burners are provided in the preheater chamber for starting-up purposes and for supplying additional heat, if necessary. H. B. T.

418. Cracking and Processing of Petroleum Hydrocarbons. J. C. Black, assr. to Gasoline Products Co. U.S.P. 1,782,676, 25.11.30. Appl., 17.5.24, renewed 17.4.30.

This patent deals with improvements on cracking processes described in U.S.P 1,426,813 and 1,456,419. The chief feature is the provision in the main furnace of a separate heater called an economiser coil. In this coil fresh charging stock or recycle oil is heated to between 600° and 700° F., just below its cracking point, and kept at such a pressure as to prevent material vaporisation. This oil is discharged into a carbon precipitating and mixing tank together with the main stream of oil from the cracking coils. This latter oil is at about 900° F., and serves to heat the economiser oil almost instantaneously to between 800° and 850° F. This instantaneous heating is claimed to increase the gasoline yield and also to exercise a marked influence on the character of the carbon formed, which is precipitated in a finely divided non-coherent form and may be readily removed from the mixing tank by blowing down. With this arrangement of heating the plant may be kept on stream four or five times as long as is normally the case. The oil leaves the mixing tank at a pressure of 1000 lb. or more per sq. in., and after passing through a heat exchanger and pressure-reducing valve, enters a separating and fractionating system. H. B. T.

419. Production of Lower Boiling Point Hydrocarbons. C. P. Dubbs, assr. to Universal Oil Products Co. U.S.P. 1,783,185, 2.12.30. Appl., 4.11.18, renewed 9.6.26.

A process and plant are described for cracking hydrocarbon oils. The oil is fed to the plant from a tank fitted with automatic level device and passed *via* heating coils to a number of horizontal vapour chambers situated in the upper part of the furnace. In these the heavy unvaporised oil is automatically maintained at a certain level, the residue being drawn off as it accumulates. The lines from the vapour chambers enter a header connected by vertical tubes with a second header. In this section of the plant the heavier fractions of the vapours are condensed and are returned to the charging line for recracking. From the upper header the light vapours are taken to a condensing and gas-separating system. In the middle of the furnace between the heating coils and the vapour chambers are situated air conduits, which serve the purpose of preheating the air supply to the burners. The plant is designed to work at pressures between 50 and 500 lb./sq. in., and the oil may be heated to temperatures between 400° and 1200° F., as required. H. B. T.

420. Cracking Hydrocarbons. G. C. Hargrove and W. B. Montgomery, assrs. Gasoline Products Co. U.S.P. 1,784,065, 9.12.30. Appl., 20.12.27.

About half an hour before shutting down a cracking plant a high level of the reflux condensate is allowed to build up in the bubble tower and the supply of fresh crude is stopped. The charging pumps drawing from the bottom of

the bubble tower are kept working so that in a short time the liquid in the tower consists of clean overhead distillate. This is forced into the heating tubes and reaction chamber until the normal fresh stock reflux mixture has been completely replaced by the clean distillate. Fires are then cut and the pump speed reduced till the oil temperature is lowered approximately 50° to 75° F. The pump speed is then further reduced, and the oil is allowed to circulate through one bank of tubes while the oil in the second bank and the reaction chamber is discharged under its own pressure to a sump. The reaction chamber is then steamed out, and when vapour free the manhole covers are removed and the chamber cleaned in the normal manner. W. L.

421. Treating Hydrocarbon Oils. J. W. Coast, Junr., and G. T. Granger. U.S.P. 1,786,053, 23.12.30. Appl., 11.12.26.

Crude oil is pumped through a closed coil in the top of a fractionating column, and is then discharged into the top of a distilling column. The light vapours pass back to the bottom of the fractionating column and the liquid is drawn off from an intermediate bubble tray and pumped at low pressure through a skimming coil. Thence it enters an expansion chamber, the liquid containing the heavy asphaltic and coke-forming constituents passing to a residue storage tank and the vapours being taken to the distilling column below the bubble trays. The condensate from the bottom of this column is pumped through a high-pressure cracking coil into an expansion chamber. The liquid which falls out passes to the residue run down line, and the cracked vapours are taken to the bottom of the distilling column. The reflux from the fractionator is fed back to one of the trays of the distilling column.

H. B. T.

422. Cracking Mineral Oil. A. E. Pew and H. Thomas, assrs. to Sun Oil Co. of Philadelphia. U.S.P. 1,786,315, 23.12.30. Appl., 24.2.26.

A process is described for cracking in vapour and liquid phase in which it is claimed little or no coke is produced. The formation of coke is due to local overheating, and to obviate this the heat is applied to the oil indirectly by means of mercury vapour. The oil can be heated to 600° F. without injurious local cracking, and this is effected in a tube preheater in the mercury boiler furnace. It then passes through a fractionating tower to a horizontal drum, which is maintained about half full of oil. From the drum the oil enters a settling chamber, and is then pumped through a nest of tubes back to the drum. The tubes are heated by means of mercury vapour supplied from a mercury boiler, and all risk of local overheating is thus avoided. From the oil drum the cracked vapours enter the fractionating tower and the reflux returns to the oil drum.

The mercury boiler may be operated at a pressure of 80 to 95 lb./sq. in., which will give a mercury vapour temperature of about 900° F. The oil temperature in the drum will then be from 750° to 800° F., while the oil leaving the mercury heat interchanger will be at about 875° F. A pressure of about 150 lb./sq. in. may be maintained on the oil system. Although no coking takes place, there is ultimately some formation of asphaltene which deposits on the tubes; this may be removed by pumping a solvent through the system. Other relatively low boiling-point metals, such as zinc or cadmium, may be used in place of mercury. H. B. T.

423. Cracking Oils. R. C. Holmes and F. T. Manley, assrs. to the Texas Co. U.S.P. 1,786,947, 9.4.26.

Oil is passed through a plurality of series connected converters, which are heated (intermediate their ends) to a uniform cracking temperature, at the same time maintaining uniform liquid levels above the heated portions of

the converters. The vapours evolved are removed and condensed, the heavier condensed constituents being returned to the first converter for retreatment. Residual oil is removed from the unheated bottom of the last converter, and a heat exchange is effected with the charging stock entering the first converter.

W. H. T.

424. Cracking Hydrocarbons. E. C. Herthel and H. L. Pelzer, assrs. to Sinclair Refining Co. of New York. U.S.P. 1,787,981, 6.1.31. Appl., 11.6.27.

The process described aims at reducing the concentration of asphaltic and pitch-like constituents in the oil in contact with the heating surfaces of the still, thus preventing to a large extent the formation and deposition of coke and increasing the time on stream. This is effected in two ways. The oil is fed through a pipe heater to what is substantially a soaking drum and back again to the heater. In the bottom of the drum are trays filled with fuller's earth or similar adsorbent earth or clay through which the oil has to pass before returning to the pipe heater. This removes the heavy asphaltic bodies and prevents their decomposition in the heating coils. The cracked vapours from the drum pass upwards to a fractionating tower, into which a certain amount of feed stock is introduced. The reflux from this tower is pumped through another coil placed in the hottest part of the furnace, and this serves to take up a certain amount of heat from the furnace gases before they come in contact with the main heating coils. The reflux oil is of a more refractory nature and can be passed through the hotter coil without deposition of carbon. After passing the second heating coil the reflux oil is discharged into the main oil stream between the first heater and the drum.

Provision is made for drawing off a portion of pitch-laden oil from above the filter bed in the soaking drum. The plant may be worked at pressures from 100 to 125 lb./sq. in., and the normal operating charge of the drum is approximately 10,000 galls., the filter bed consisting of four to seven tons of fuller's earth preferably of 15 to 30-mesh. The temperature maintained in the drum may be from 750° to 775° F., and the oil discharged from the second heating coil may be at a temperature of 775° to 825° F. Charging stock may be pumped into the feed lines of either of the pipe heaters. H. B. T.

See also Abstracts Nos. 412, 415.

Hydrogenation.

425. Hydrogen from Methane and Similar Hydrocarbons. D. Tyrer and Imperial Chemical Industries, Ltd. E.P. 340,050, 3.10.29.

Air is supplied to a deep bed of coke at a number of points at different depths of the bed, and hydrocarbon gas, such as coke oven and other gas containing methane, with or without steam, is alternately supplied. Preferably the hydrocarbon traverses the whole depth of the coke bed in an upward direction and opposite to the air blast. The hydrocarbons are cracked in the hot coke and much of the carbon is deposited, thus serving to supplement the coke burnt in the blow period. The maximum amount of air is supplied near the point of exit of the blow gases.

In cases where the hydrocarbon gas is substantially pure the process is self-supporting as regards carbon, and no coke need be added. The bed of coke may therefore be replaced by a bed of inert material. When steam is added, however, coke is necessary to meet the requirements of the water gas reaction. R. P.

426. Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind. A.-G. E.P. 340,470, 29.12.30.

Acid tars, obtained in refining mineral oils or benzol by means of sulphuric acid, or the solid products arising from the condensation of middle oils in the presence of aluminium chloride or other metal halides, have hitherto been worthless.

Valuable liquid hydrocarbons may be produced from these substances by treatment with desulphurising or neutralising agents, such as Florida earth, lime, or iron oxide and subjecting them to destructive hydrogenation. It is advantageous to mix these residues with an oil such as tar oil before hydrogenation.

The process is carried out under pressure, and when catalysts are employed these should be immune to sulphur poisoning. Oxides of molybdenum, tungsten and chromium, or cobalt sulphide are suitable. Temperatures between 300° and 700° C. are employed, preferably between 370° and 500° C. R. P.

427. Destructive Hydrogenation of Carbonaceous Materials. Standard Oil Development Co. E.P. 340,811, 8.1.31. Conv. (U.S.A.), 22.1.29.

Finely ground carbonaceous material is suspended in heavy oil and pumped through a heating coil into a reaction chamber constructed to withstand high pressure. The contents of the chamber are agitated by means of a stirrer and the temperature is maintained in a suitable manner. A part of the charge is continuously withdrawn from the bottom of the reactor and circulated back through the heating coil. Hydrogen is forced into the stream of oil before entering the coil. Vapours from the reaction vessel pass through heat exchangers to condensers and gas separators. The gas is purified and returned into the process whilst a proportion of the charge is withdrawn continuously from the reaction vessel into a suitable chamber which is maintained at a pressure sufficient only to allow the oil to be vaporised and separated from the ash and residue. R. P.

428. Destructive Hydrogenation. B. R. Goodfellow, L. Patrick, K. Gordon and Imperial Chemical Industries. E.P. 341,069, 14.12.29.

In hydrogenation of coal and oil it is not essential to use pure hydrogen. Hydrogen, containing hydrocarbon and other gases may be employed. This invention uses the hydrogenating gas cyclically in such a manner that there is no accumulation of uncondensable gases, ethane, methane, or nitrogen. The gases are therefore treated at one or more points in the circuit for the removal of the inert gases, which removal need not be complete.

Partial removal is effected by cooling the gases to a low temperature (−190° C.) after the liquid products have been condensed and separated. The operation is carried out at a pressure of 20 atm. Before cooling, make up gas, *i.e.*, purified coke oven gas, is added to and cooled along with the gas leaving the converter, in order to remove nitrogen and other unwanted constituents. The gases may be cooled to liquefy the ethane, but not the methane and nitrogen; then the residual gas is washed with the liquefied ethane. Practically all the methane and some of the nitrogen is dissolved. By this method the gas returning to the converters contains at least 90% of hydrogen. R. P.

429. Reactions with Hydrogen. I. G. Farbenind. A.-G. E.P. 341,153, 15.1.31.

In the treatment of coals or oils with hydrogen suitable constructional materials are difficult to find. Iron is decarbonised by hydrogen and becomes brittle, whilst sulphur and sulphur compounds have a corrosive

effect. It has been found that zinc and zinc alloys, such as brass, are impermeable to hydrogen, and in contrast to copper are not attacked by sulphur compounds.

Alloys of this nature are therefore suitable for the construction or lining of plant used in hydrogenation. Nickel and zinc; iron, nickel and zinc; copper, iron, and zinc; and the like, are also suitable provided they do not melt at the working temperature. Ammonia, water, phenols and sulphuretted hydrogen have no effect on these alloys. Again, these alloys do not cause the deposition of carbon or the formation of methane. Further it has been found that the concentration of hydrogen is preferably regulated according to the amount of sulphur present in the raw material; the higher the sulphur content the greater should be the concentration of hydrogen and so adjusted that not more than 2 to 3% of hydrogen sulphide is present in the gases.

An apparatus is described wherein these alloys are utilised in the construction. R. P.

Chemistry of Petroleum.

430. Viscosity Dilution. J. Tausz and A. Rabl. *Petr. Zeit.*, 1931, **27** (3), 41–43.

The value of x in the expression η_x undiluted$=\eta$ diluted is a measure of the viscosity dilution where η is expressed in centipoises and the oil is diluted by means of a solvent. Results are given of the viscosity dilution measurements of mineral oils, fatty oils, fatty acids, products of industrial refining and chemically pure substances.

The viscosities were determined in an Ostwald viscometer placed in an accurate thermostat at the experimental temperature and at atmospheric pressure. The values of x for various commercial oils are given. The index x indicates the content of the dilution medium (toluol) as a percentage by weight in the oil mixture.

The results show generally that the change in viscosity of a substance on dilution depends on its chemical composition. An association of the oil molecule is assumed with mineral oils, and this is in agreement with the viscosity dilution curves. A rather extensive adsorption of the dilution medium by the oil molecule occurs in fatty oils. Aromatic compounds have a low value for x, naphthenes have a high value for x. The value of x increases in oxygen-containing compounds according to the quantity of oxygen and in its mode of union in the molecule. The effect of carboxyl groups, *e.g.*, in fatty acids, is to increase x.

The work is elaborated with tables giving the values of η undiluted, η_{10}, η_{20}, x_{10}, x_{20}, etc., for the substances mentioned, η being expressed in centipoises. W. S. E. C.

431. Study of Mercaptan Chemistry. W. M. Malisoff, E. A. Marks and F. G. Hess. *Chem. Rev.*, 1930, (4), 7, 493–547.

A review of the field of mercaptan chemistry is given, the term "mercaptan" denoting any compounds containing one or more —SH groups. Besides giving a critical presentation of the accumulated data, the results of such a presentation are discussed and outstanding research problems outlined. The subject has been treated under the headings of physical characteristics, chemical characteristics, method of preparation and purification, detection and determination, special interest attaching to the substances and research problems. References to mercaptan chemistry in the literature are tabulated. W. D. S.

432. Action of Alkali Hydroxides on Elementary Sulphur and Mercaptans dissolved in Naphtha. V. Vesselovsky and V. Kalichevsky. *Ind. Eng. Chem.*, 1931, **23**, 181–184.

A study has been made of the effect of sodium and potassium hydroxides in various solvents on solutions of elementary sulphur and mercaptans in naphtha, in connection with the possibility of using the reactions in refining petroleum distillates. The solvents employed were ether, ethyl alcohol, isopropyl alcohol and water.

On adding the reagent to a solution of elementary sulphur in naphtha, a yellow precipitate is formed and the oil becomes yellow. Sulphur is removed with the formation of alkali thiosulphate and trisulphide. If the trisulphide is kept in suspension in the naphtha, penta- or hexa-sulphides may be formed. After all the elementary sulphur has been removed, agitation in the presence of air may result in some of it being returned to the naphtha owing to its reformation by the action of air on thiosulphate or polysulphides. These actions occur more readily with potassium than with sodium hydroxide. Aqueous solutions of hydroxides react similarly but very slowly.

When solutions of alkali in absolute alcohols are added to naphtha solutions of ethyl, n-butyl and n-heptyl-mercaptans, a dark-brown liquid separates out. The naphtha is sweetened completely by removal of the mercaptans as alkali mercaptides. Sodium hydroxide acts more rapidly than potassium hydroxide in this case. E. B. E.

433. Catalytic Reduction of Carbon Monoxide under Ordinary Pressure. VI. The Hydrocarbon-forming Action of Iron Catalysts. S. Kodama. *Sci. Papers Inst. Phys. Chem. Res. Tokyo*, 1930, **14**, No. 269, 169–183.

Iron catalysts for synthetic hydrocarbons are of greater commercial importance than those containing cobalt. No experimental details on iron catalysts have yet appeared. Iron catalysts produce no water; on the other hand cobalt catalysts invariably produce water, sometimes several times as much as hydrocarbon. The carbon dioxide content of the reaction gas is about 20% when using iron catalysts; with cobalt catalysts 5–10%. The unconverted reaction gas with an iron catalyst contains more carbon monoxide than hydrogen; the reverse occurs with cobalt catalysts. Iron catalysts give a coloured oil, cobalt catalysts seldom produce a yellow and never a coloured benzene. Iron catalysts require a higher temperature than those of cobalt. Catalysts containing mostly iron withdraw oxygen from the carbon monoxide in the form of carbon dioxide. Since carbon monoxide is more decomposed than hydrogen the hydrogen content of the final gas is always greater than the carbon monoxide content; with cobalt *vice versa*. A catalyst containing iron 1 pt., copper 1 pt., sodium carbonate 0·02 pts. gives the best yield, 1·8 c.c. of kerosine and 1·4 c.c. of benzene from 157·0 litres of gas which is lower than that from cobalt-copper catalysts containing magnesium oxide or zirconium oxide. The addition of magnesium oxide was detrimental to the iron catalyst. Without addition of alkali an iron-copper catalyst forms only lower hydrocarbons. W. S. E. C.

434. Heats of Combustion of Methane and Carbon Monoxide. F. D. Rossini. *Bur. Stand J. Res.*, 1931, **6**, 37–49.

These determinations have been carried out as there is some uncertainty attached to the usually accepted values obtained by Thomsen over 50 years ago.

The apparatus consists essentially of a calorimeter containing a specially designed reaction chamber and a heating coil.

A quantity of the purified gas, measured by determining the mass of the reaction products, is burned in an excess of oxygen in the reaction vessel and observations of temperatures are made during a preliminary, reaction and after period.

The apparatus is restored to its initial temperature conditions and an electric current passed through the heating coil so that the heating effects of the combustion experiment are exactly duplicated.

The electrical energy thus required is accurately measured and is equal to the heat of combustion of a known quantity of gas.

The heat of combustion of CH_4 to form liquid H_2O and gaseous CO_2 is found to be $889{,}700 \pm 300$ international joules per mol. at 30° C. and a constant pressure of 1 atm., which is equivalent to $890{,}160 \pm 300$ international joules per mol. at 25° C., 1 atm.

The heat of combustion of CO to form CO_2 is found to be $282{,}925 \pm 120$ international joules per mol. at 30° C. and 1 atm., which is equivalent to $282{,}890 \pm 120$ international joules per mol. at 25° C. and 1 atm.

Comparisons are made with former work carried out on this subject.

D. L. S.

435. Isolation of the Isomers of Hexane from Petroleum. J. H. Bruun and M. M. Hicks-Bruun. *Bur. Stand. J. Res.*, 1930, **5**, 933–942.

On ordinary fractional distillation of an Oklahoma crude oil the hexanes were concentrated in a set of constant-boiling mixtures, the other constituents of which were ring compounds. These mixtures were broken up by distillation after the addition of an alcohol which was later removed by water washing and, by the aid of equilibrium meeting, four of the five isomers of hexane were obtained and identified: 2, 3-dimethylbutane, 2-methylpentane, 3-methylpentane, and *n*-hexane; 2-2-dimethylbutane was not found.

The following constants were determined for each hexane: B. pt., freezing pt., sp. gr. and refractive index. The freezing points found for two of the hexanes appear to be the only ones on record. They are, for the air-saturated hydrocarbon, $-143 \pm 0{\cdot}5$° C. for 2-methylpentane and $-118 \pm 0{\cdot}5$° C. for 3-methylpentane. The eutectic for the system CO_2-2-2 methylpentane is $-153 \pm 0{\cdot}5$° C.

G. S.

436. Synthesis of 1, 4—Pentadiene. P. N. Kogerman. *J. Amer. Chem. Soc.*, 1930, **52**, 5060.

An improved method is described for the preparation of vinyl bromide from an alcoholic solution of potassium hydroxide, ethyl alcohol and ethylene bromide. The yield of vinyl bromide obtained was 85% of the theoretical, the vinyl bromide so obtained being dry and pure, having b.p. 16-17°C.

Two modifications of a new method for the preparation of 1, 4—pentadiene from dry ether, magnesium, vinyl bromide and allyl bromide are described, namely, (*a*) under atmospheric pressure and (*b*) under super-atmospheric pressure. In both, the 1, 4-pentadiene was obtained as its tetrabromide, and the hydrocarbon was finally regenerated from the tetrabromide. The total amount of 1, 4-pentadiene recovered from the pure tetrabromide, which had m.p. 85-86°C. and. mol. wt. 376, was about 73% of the theoretical. The hydrocarbon so obtained was finally fractionated through a Widmer column, and had b.p. 25·8-26·20/756 m.m.; d^{20}_4 0·6594; n^{20}_g1·3883; mol. refraction; calcl., 24·26; found 24·26; mol. wt. (Victor Meyer method); calcl., 68·08; found 68·66.

The bromination products of 1,4-pentadiene were investigated. H. E. T.

437. Slow Combustion of Ethane. W. A. Bone and S. G. Hill. *Proc. Roy. Soc.*, 1930. *A*. **129**, 434, 457.

The "hydroxylation" and "peroxidation" theories of hydrocarbon combustion are discussed. Experiments have been carried out to determine if a peroxide is formed during the slow combustion of ethane, and if so, at what stage in the process. The investigation covered a temperature range of 290-323° and initial pressures between 440 and 780 m.m. The results indicate that the oxidation proceeds homogeneously throughout the medium, being greatly retarded by increasing the surface exposed to the gases, that it is preceded by a well-marked induction period, during which no oxidation occurs, and that it is accompanied by a steady pressure rise, and the production chiefly of aldehydes, steam and oxides of carbon, but of neither hydrogen nor free carbon. The induction period can be considerably reduced by the addition of small amounts (say 1%) of moisture, iodine, nitrogen peroxide, ethyl alcohol, formaldehyde, etc., to a dry $C_2H_6+O_2$ medium without altering the non-explosive nature of the combustion. The addition of 1% of acetaldehyde, however, causes an instantaneous inflammation of the medium. A rise of temperature or an increase of pressure shortens the induction period; a rise of temperature also shortens the "reaction period," and an increase in pressure tends to shorten it. Both the induction and the reaction periods are much shorter for a $(2C_2H_6+O_2)$ than for a $(C_2H_6+O_2)$ mixture; and are rapidly lengthened by an increase in oxygen content beyond an equimolecular proportion. This is consistent with the "hydroxylation," but not with the "peroxidation," theory. Although inconsiderable amounts of a "peroxide" appear to be formed at the acetaldehyde stage of the combustion, no evidence was obtained of any earlier peroxide formation. Indirect experimental evidence supports the view that ethyl alcohol or some lower "oxyethane," rather than $C_2H_6O_2$, is primarily formed in the slow combustion of ethane. W. S. E. C.

438. Heat of Hydrogenation of Ethylene. H. Von Wartenborg and G. Kranse. *Z. Physikal Chem.*, 1930, **151**, 105, 108.

Calorimetric measurements lead to the value $30{\cdot}0 \pm 0{\cdot}3$ kg.-cal. for the reaction $C_2H_4+H_2=C_2H_6$ at constant volume. Hydrogenation was effected with palladium sol by Skita's method. W. S. E. C.

439. Velocity of Polymerisation. I.—Polymerisation of Diethylenic Hydrocarbons. II.—Polymerisation of Isoprene. E. N. Gapon. *J. Russ. Phys. Chem. Soc.*, 1930, **62**, 1385–1393, 1395–1406. (Br. Chem. Abs. 1931, p. 45.)

I. A theoretical consideration of the polymerisation of diethylenic compounds indicates that the activation of substrate molecules depends on the opening of a double linking with the formation of tervalent carbon. The energy required represents the energy of activation, the polymeride being formed by a combination of active with inactive molecules. II. The ratios of the values of the velocity coefficients of the reaction of polymerisation of isoprene to dipentene, to 1 : 3-dimethyl-3-ethenyl-Δ^1'*cyclo* hexene, and to the polymeride are 530 : 41 : 1. The variety of products may be explained by the three types of activated molecules being formed in the polymerisation of isoprene. The temperature coefficients of the above reactions diminish in the order given as do the heats of activation and the activity constants of the active molecules. W. H. T.

440. Properties of Conjugated Compounds. Part XI. Addition of Hydrogen Bromide to $\beta\gamma$ and $\alpha\delta$ Dimethyl Butadiene. E. H. Farmer and F. C. B. Marshall. *J.C.S.*, 1931, **134**, 129–137.

The work of Bergmann (*J. Russ. Phys. Chem. Soc.*, 1920, **53**, 37) and Claisen (*J. Prakt. Chem.*, 1922, **105**, 65) on the addition of hydrogen bromide to $\beta\gamma$ dimethyl butadiene is reviewed. The authors conclude from their own experiments that addition takes place solely in the $\alpha\delta$ position.

In the addition of hydrogen bromide to $\alpha\delta$ dimethyl butadiene both $\alpha\beta$ and $\alpha\delta$ addition products are formed since a mixture of glycols is obtained by gentle permanganate oxidation, and this in turn is convertible to a mixture of mono and dibasic acids.

The absence of methyl succinic acid among the products shows that $\alpha\beta$ addition takes place in only one of two possible ways. From the relative proportions of acetic and propionic acids it is estimated that the $\alpha\beta$ hydrobromide forms 90% of the product. A. J. G.

441. Action of Reduced Nickel and Hydrogen on Aromatic Hydrocarbons under High Pressure and Temperature. T. Ogawa and T. Yokota. *Bull. Chem. Soc. Japan*, 1930, **5**, 266.

100 gm. benzene and 5 gm. reduced Ni and H_2 were heated for 1 hour at 470° under a pressure of 80 atm. at 0°, the reaction showing a depression of H_2 of 35 atm. at 0°. The gas in the autoclave contained 98·5% H_2, 1·2% N_2, 1·4% satd. hydrocarbons and 0·1% CO., the liquid reaction product (92%) being cyclohexane, which is stable under the above conditions. Benzene and Ni heated with H_2 at 500° and 100 atm. gave 28% gas, 41% liquid hydrocarbons and 14% coke. The gas consisted of 94·5% CH_4 and 5·5% H_2, and liquid (b.p. 48°-320°) C_6H_6 and Ph. Me. Benzene at 200° and 100 atm. for 2 hours and then at 500° for 3 hours gave 5·4% CH_4 and a mixture of cyclopentane, methylcyclopentane and cyclohexane. Pure $C_{10}H_8$ heated with H_2 at 460°-500° and 80 atm. with or without Fe_2O_3 or CuO showed no reaction, but with Ni there resulted 5% CH_4 and 91% of a liquid composed of benzene homologues and tetralin. The latter treated as in the case of $C_{10}H_8$ gave 5·6% CH_4 and 92% of a liquid consisting of benzene homologues and decalin. Decalin gave cyclohexane and its homologues. W. H. T.

442. Synthesis of Some Higher Paraffins. S. Lander and R. Riedl. *Coll. Czechoslav. Chem. Comm.*, 1930, **2**, 520.

The following reactions have been used to prepare $Me(CH_2)_{14}CHMe_2$ (I) and $Me(CH_2)_{20}CHMe_2$(II). The ester with two less C atoms with Me Mg Br gave an alcohol yielding on distillation two olefins, the latter being hydrogenated to give the paraffin.

$Me(CH_2)_{14}CMe_2OH$ was formed from Et. palmitate (yield 90%) a 20% excess of Me Mg Br being used. The alc. was dehydrated by distillation at 100 mm., 2. Methyl-2-heptadecene (m.p.-2·5°, b.p. 314°, d_{20} 0·7953) being the only product. Hydrogenation was carried out at 220°-240° for 82 hours, the unchanged olefin brominated and the two products separated by distillation. Rape oil was hydrogenated, using 2% Ni. at 120 atm., the solid product being saponified with twice the theoretical amount of alc. KOH., and the acid freed with dil. H_2SO_4. The acid, after heating to 125° was dissolved in alcohol, HCl gas passed in, water added and the product heated to 135°. 20% Et. butanate was yielded by distillation at 15 mm. and 252-8°, recrystillation from alcohol and a final distillation at 255°. Treatment with twice the theoretical amount of Me Mg Br, distillation at 0.002 mm., and crystillation from Et.$_2$O yielded 90%. $Me(CH_2)_{20}CMe_2OH$(m.p.63°). Distillation of the alcohol at 290° and 100mm. yielded $Me(CH_2)_{15}CH{:}CMe_2$

(m.p.41·5°, C.p. 234·7°). Hydrogenation carried out at 180° and 1 atm. was incomplete, and the product was obtained by twice precipitating an Et_2O solution with half its volume of alcohol. The product had an iodine no. 1·2, m.p. 42°, d 0·7882 at the m.p. W. H. T.

Analysis and Testing.

443. Apparatus and Method for Analysis of Gas Mixtures by Combustion and Absorption. M. Shepherd. *Bur. Stand. J. Res.*, 1931, **6**, 121–167.

This volumetric apparatus for the analysis of gases is of the "Orsat" type and a detailed description is given of each separate unit together with full instructions for assembling. Among many modifications are specially designed absorption and combustion pipettes and a manometer compensator unit giving an improved control of the pressure balance. Detailed instructions are given for the carrying out of an analysis. The apparatus has been found to give more accurate results and to be easier to manipulate than many other models. D. L. S.

444. Analysis of Mixtures of Hydrogen, Methane and Ethane. E. M. J. Mulders and F. E. C. Scheffer. *Rec. Trav. Chim.*, 1930, **49**, 1057–1065.

A phase-rule study of the system hydrogen-methane-ethane at the temperature of liquid air (–190°), which is above the critical temperature of hydrogen, shows that separation of the mixture of these three gases into a solid phase (ethane) and a gaseous phase (hydrogen and methane) is effected at this temperature under diminished pressure, and an apparatus for the accurate analysis of such mixtures on this principle is described in detail. Some methane is occluded by the solid ethane, the quantity being smaller when hydrogen is present, but larger in the absence of hydrogen, since condensation is more rapid in this case. The two fractions obtained in this manner are then separately analyzed by oxidation with hot copper oxide, the water and carbon dioxide formed are condensed in liquid air, and the volume of any inert gas is determined. The water is then absorbed by phosphoric oxide, and the carbon dioxide collected and measured. From the data thus obtained the composition of the original mixture may be calculated. In the apparatus described the whole determination may be effected in two hours, and the accuracy of the method is proved by analysis of mixtures of known composition. W. S. E. C.

445. Determination of Hydrogen Sulphide in Refinery Still Gases. A. R. Schamagel and A. W. Trusty. *Ind. Eng. Chem. Anal. Edn.*, 1931, **3**, (1), 29

The iodine titration method for the determination of hydrogen sulphide in still gases is inaccurate because of the reaction of the gaseous unsaturated hydrocarbons with the iodine. The authors have therefore developed the following method: 100 c.c. of 0·1N acid lead nitrate is placed in a Fischer gas washing bottle and a metered quantity of the gas passed through. A second similar bottle is placed in series with the first and with the slightest discoloration of the second, the flow of the gas is reduced to insure all of the hydrogen sulphide being caught in the first bottle. The contents of the first bottle are finally filtered, made up to volume, and aliquot portions titrated with ammonium molybdate solution. W. H. T.

446. Manometer for Measurement of Small Pressure Differentials at High Pressures. J. H. Boyd. *J. Amer. Chem. Soc.*, 1930, **52**, 5102.

The manometer described will measure a pressure differential of 1 in. of mercury at a pressure of 3000 lb/sq. in. with an accuracy of one part in a thousand. The apparatus consists of a relatively large diameter mercury reservoir beneath whose surface projects a relatively small diameter riser well in which a moveable contact piece is placed so that the mercury level is determined by the height of a projecting rod attached to the contact piece, electrical contact being made when the contact touches the mercury. Connections of downstream and upstream gases are made to the main and riser well respectively, and pressure differentials read off directly from the projecting rod height relative to the contact height when no gas flows. The use of chemically clean mercury is not absolutely necessary, but it is advisable.

A. R. S.

447. Electrically heated Melting Point Apparatus. C. E. Sands. *Ind. Eng. Chem. Anal. Edn.*, 1931, **3** (1), 65.

The apparatus consists of an electrically heated modified Thiele-Dennis m. pt. tube, a lead container filled with sand to prevent damage by sulphuric acid in case of breakage, a lamp and a rheostat, all suitably mounted for convenience and portability. The m. pt. tube (of Pyrex) has a smaller tube closed at the lower end sealed within in the vertical position and extending 1-2 ins. below the point of attachment of the side arm. The side arm is wound at its lowest portion with an asbestos covered chromel heating element, and insulated with asbestos which, with a portion of the vertical tube, is painted with aluminium paint to minimise radiation. The use of a mixture of sulphuric acid and potassium bisulphate as the heating liquid makes it possible to reach a temperature of about 300° C., provided that the absorption of moisture by the liquid is prevented by means of a calcium chloride tube when the apparatus is not in use. With a current of 1·4 amps. the temperature in the inner tube is raised to 100° C. in about 6 min to 200° C. in 12 min, and to 300° C. in 25 min. The rapidity of change from one temperature to another is regulated by means of the adjustable rheostat. The melting of the substance is observed by the aid of light from the lamp coming through a small hole in a suitable screen.

W. H. T.

448. Potentiometric Determination of Saponification Number of Mixtures of Asphalt and Drying Oils. J. V. Demarest and W. Riesman. *Ind. Eng. Chem. Anal. Edn.*, 1931, **3** (1), p. 15.

The determination of the saponification number of mixtures of asphalt and drying oil is of considerable importance in the testing of asphalt paints. The standard method is difficult and inaccurate because the black colour of the asphalt suspension obscures the end point of the indicator. When the benzene is evaporated considerable alkali is occluded by the asphalt. The method described overcomes the first difficulty by the use of the hydrogen electrode, and the second difficulty by the use of anisole as a solvent. To a weighed sample, 50 c.c. of standard alcoholic KOH and 40 c.c. of anisole are added, and the mixture refluxed until saponification is complete. After cooling it is titrated potentiometrically with standard aqueous hydrochloric acid, normal calomel and hydrogen electrodes being used. After each titration the hydrogen electrode is treated with hot conc. HNO_3, and then made the cathode in the electrolysis of dilute sulphuric acid, after which it is ready for the next titration. Anisole was found to be the most suitable solvent for the purpose; ethyl ether, propyl ether, isopropyl ether failed to keep a major portion of the asphalt in solution. Nujol formed a thick emulsion when

hydrogen was bubbled through saponified mixtures of linseed oil and asphalt, and benzene poisoned the hydrogen electrode. During the titration, two layers separate, the upper anisole layer containing almost all of the asphalt, 9 mins. being required to attain a steady potential after each addition of acid. W. H. T.

449. Distillation Test Innovation Indicates Wide Utility Range. R. C. Conine. *Oil & Gas J.*, 1931, **29** (36), 105, 107.

The chemists of the Carter Oil Co., in conjunction with engineers of the Tulso Refrigeration Co., have perfected a new method for carrying out the distillation test. The charged flask is heated by electric coils, and the condensers and receiving jar baths are cooled by the Frigidaire system. A new cooling medium which is non-corrosive and constant in composition is employed, and circulated to the condensers and receivers at the rate of 6-gal./min. by means of a centrifugal pump. The condensers are insulated and sealed, and the pyrex glass receiving jars can be adjusted to receive a beaker or graduate. Temperatures are regulated by valves within the range 32° to 40° F. normally employed for distillation of ordinary and natural gasolines, but by adjusting the compressor unit a range of −4° to −10° F. can be obtained. The apparatus is capable of running four tests simultaneously. The advantages claimed are: (1) Saving of time in preparing apparatus for test. (2) Distillation loss more constant. (3) Cost of ice eliminated. (4) Apparatus is easily adaptable for other uses, such as cold test determinations. Specifications for the Frigidaire cabinet, receiving jars, centrifugal pump, compressor equipment, cooling tank and electrical equipment are given. R. A. E.

450. Vacuum Fractionation of Phlegmatic Liquids. K. Hickman and W. Weyerts. *J. Amer. Chem. Soc.*, 1930, **52**, 4715.

The word phlegmatic is used to describe those liquids of high boiling point. It is shown that whereas it is ordinarily assumed that in a low-pressure distillation pressures are constant at all parts of the apparatus at a particular moment, this is not actually the case. In order to measure the actual pressure in the space surrounding the thermometer, the latter is placed in a large bulb made at the top of the fractionating column where the vapour velocity is low, and a side arm is fitted to this bulb which goes directly to the manometer. A description of a direct reading oil manometer used is given as an appendix to the paper. A. R. S.

451. Estimation of Asphalt. J. Givaudon. *Rev. Pétrolifère*, 1931, No. 408, 105.

The author deals with the estimation of hard asphalt using petroleum ether as the precipitant. There are three types of petroleum ether in current use: (1) A spirit (Kahlbaum) b. pt. 65°–95° C., sp. gr. 0·695–0·705 with less than 2% soluble in sulphuric acid (80% C.O.V., 20% oleum). This material contains about 75% of hexane and traces of benzene. (2) The material specified by the I.P.T., b. pt. (90%) 60°–80° C, sp. gr. 0·680–0·690, aromatic content 0·5%. (3) Petroleum ether (Chemins de fer français) b. pt. 35° C.–50° C., sp. gr. 0·640–0·645. This material is principally pentane and is free from aromatics. It is obvious that the final result will be affected by the type of precipitant used and that having decided upon the type of spirit, the method of operation must be standardised. After dealing with the various aspects of the estimation and its manipulation, the author describes the following procedure which he has adopted: 4 gr. of the oil are weighed into a conical Erlenmeyer flask and dissolved in 200 c.c. of petroleum ether, then being allowed to stand in the dark for 48 hours. During this time the filter is

prepared. 3 to 4 g. of powdered asbestos are formed into a suitable pack in a sintered glass (Gooch) crucible. The filter is washed (using a vacuum) with pet. ether and benzene in order to remove traces of oil. The filter is dried at 100° C. and tared in a weighing bottle. When precipitation is complete, the asphalt is filtered off in the prepared filter (using vacuum), washed with pet. ether, dried at 100° C., and finally weighed. The filter is then replaced in the vacuum flask, and the asphalt washed with boiling benzene. The filter is again dried and weighed, the two weights giving the quantities of asphalt soluble and insoluble in benzene. As an additional check, the benzene solution may be evaporated and the residue weighed. W. H. T.

452. Viscosity Determinations at Low Temperatures. E. Schlenker. *Brennstoff-Chem.*, 1930, **12** (2), 25–26.

Vogel's U-tube apparatus is described. It consists of a closed vessel half filled with water and connected by tubing to a pipette drawn out at one end to a capillary, whose tip is immersed in an oil absorption vessel. The latter is surrounded by a double-walled metal case, the outer case containing ice and the inner case an ice and salt mixture. The water flowing out of the outlet tube of the water-vessel effects the oil-level in the pipette and ceases to flow when the difference in the level (h) between the surface of the water and the lower end of the outlet tube is equal to the quotient of h and the sp. gr. of the sample. In the condition of equilibrium the following equation is satisfied :—

$$\frac{h}{10} = 1 - p \text{ and } \frac{V_0}{V} = \frac{p}{p_0}$$

If $p_0 = 1$, $h = 10\left(1 - \frac{V_0}{V}\right)$ where :—

h = desired increase in height of liquid in metres.

V_0 = volume of void space in the water vessel before the discharge of the water.

V = corresponding volume under conditions of equilibrium.

p_0 = air pressure in atmospheres before the discharge of the water (= 1).

p = air pressure in atmospheres under conditions of equilibrium.

The h is adjusted so that an easy removal of the oil between both marks on the pipette is possible ; the outlet tube h from the water vessel can be extended in order to increase the velocity of outflow.

Experiments were carried out with an American refined spindle oil. At present the apparatus has been calibrated, so that a time increase of 50 secs. corresponds to 7° E. W. S. E. C.

453. Viscosity Data in Graphical Form. R. P. Genereaux. *Ind. Eng. Chem.*, 1930, **22**, 1382.

Charts of viscosities plotted by the method of Ravenscroft are given, all values being in centipoises. Values are plotted on log-log paper against absolute temperature. The points for most of the fluids given lie along straight lines, but in cases of deviation the nearest straight line is taken.

In the gas viscosity chart two logarithmically scaled lines of temperature and viscosity are placed parallel and close to each other. Temperature and viscosity values were taken for two points from the straight line to represent the variation of viscosity with temperature for each gas. Lines are then drawn through the corresponding values on the temperature and viscosity lines on the chart. The intersection of these lines determined a point charac-

teristic of the fluid. A line from a point on the chart drawn through a temperature value will cross the viscosity line at the value for that temperature.

The graph for liquids follows the same procedure, but in this case the points fell between the viscosity and temperature scales, because liquids decrease in viscosity with rise in temperature. All data are for atmospheric pressure.

A. R. S.

See also Abstracts Nos. 370, 372.

Engines.

454. Effect of Length-Diameter Ratio of Oil-Spray Orifice is Investigated. A. G. Gelalles. *Aut. Ind.* 1931, **64**, 116–117.

Nozzle orifices of 0·014 in. and 0·040 in. were used in the tests, and the length/diameter ratio was varied between 0·5 and 4·0. The nozzles were mounted in an injection valve and tested with a plain and helical-grooved stem. Injection pressure varied from 4000–8000 lb/sq. in.

Using a high grade Diesel fuel (sp. gr. 0·86) with a plain stem, a minimum penetration is reached with a ratio between 1·0 and 2·5, while it attains a maximum at 3·5 and over. Increase in the air density into which the fuel is sprayed decreases the rate of change of penetration with the length/diameter ratio. With a helical grooved stem an increase in the length/diameter ratio first causes an increase in penetration, but as the time is increased a minimum occurs at a ratio between 0·5 and 2·0 with an 0·014 in. orifice. As the orifice diameter increases, the tendency for a minimum penetration to occur is lessened. With centrifugal sprays the penetration was much less than with plain sprays, owing to the tangential velocity component. As the length/diameter ratio of the orifice increases so this component is decreased with a resultant increase in penetration. With a stem of this type the coefficient of discharge is about 2·8 times as great with the 0·014 in. nozzle as with the 0·040 in.

C. C.

455. The Origin and Development of Heavy-Oil Aero Engines. D. R. Pye. *Engineer*, 1930, **150**, 705.

Elimination of electrical ignition, use of a cheaper fuel at a higher thermal efficiency, and the reduction of fire risks are the principal advantages of the heavy-oil over the petrol engine. For aero work, however, the weight-power ratio is also of prime importance. To compete with the petrol engine, weighing 2 lb or less per B.H.P., the heavy-oil engine must be cut down to about 3½ lb/B.H.P. This figure is based on weight of engine and fuel for a 10-hour flight, taking the fuel consumption as 0·4 lb and 0·55 lb/B.H.P.-hour for the heavy-oil and petrol engine respectively.

Three different types of heavy-oil engine have reached a practical stage of development at the present time. Of these, the Beardmore, 8-cyl. in line, water-cooled engine is still unduly heavy, but certain practicable modifications should reduce the weight per h.p. to less than 4 lb. The other two engines, the Junkers SLI, 6-cyl. opposed-piston 2-str. at 3·08 lb/B.H.P., and the Packard 9-cyl. air-cooled radial at 2·26 lb/B.H.P. are well within the limit. Experience is lacking of the sleeve valve type of engine, now being developed by H. R. Ricardo. The design has great possibilities, however, in view of the control of turbulence it can give. The same advantage lies in the use of supercharging, the possibilities of which for compression ignition have hardly yet been explored.

G. B. M.

General.

456. Petroleum Exploitation in the Bacau District. B. Sergescu. *Ann. Min. Roumanie*, 1931, **14** (2), 50–56.

This article deals firstly with the exploitation of oil deposits near Dofteana by ordinary mining methods. Two galleries have been driven into the Oligocene and Eocene at angles of about 30°, and one shaft has been sunk to a depth of 168 metres. The method of constructing the shaft is given in some detail. Work has now been abandoned although moderate supplies of oil were actually obtained.

In the second section the writer describes the Campeni area where 150 shallow wells have been put down. Production in this area is diminishing, but it is claimed that there is a considerable chance of deeper oil sources than those already tapped. V. C. I.

457. An Air-Thermostat for Corrosion Research. U. R. Evans. *J.S.C.I.*, 1931, **50**, 66–67.

The ordinary form of air-thermostat used for research on the corrosion of metals introduces inaccuracies, as the temperature assumed by any solid body placed in an air-thermostat is a "compromise temperature," i.e., the value which permits it to receive heat by radiation or convection from the heater just as quickly as it loses heat to the walls or to the open air.

The only way to guarantee true identity of temperature of the specimen, thermo-regulator and thermometer is to ensure that the "walls" of the enclosure are themselves at the chosen temperature. This is best attained by using a double-walled cupboard, the inner walls being of metal and the outer walls of insulating material; the air is circulated in the passages between the walls, which are thus brought to the correct temperature; the heaters and coolers should be placed in the passages, the inner walls being lagged at these points so as to prevent direct radiation to the experimental vessels.

Complete details of the suggested air-thermostat are given and it is found that disturbing effects of thermal convection currents, which are inevitable if the vessels receive heat on one side and lose it on the other, are avoided.

The temperature is controlled by an adjustable contact thermometer working a "B.G. & B." thermionic valve relay fitted with a mercury switch.

It is suggested that some of the features of this cupboard may have application for purposes other than corrosion research. W. A. W.

458. Viscous Flow and Surface Films. R. Bulkley. *Bur. Stand. J. Res.*, 1931, **6**, 89–112.

Experiments have been carried out on the flow of various liquids through fine glass and platinum capillaries in an attempt to detect any departure from the laws of simple viscous flow.

Previous workers have observed that in many cases liquids flowing through capillaries have gradually clogged the tubes and have pictured the progressive building up of an immobile layer on the inside of the capillary due to orientation and selective adsorption of polar constituents.

The author shows that clogging is due entirely to extraneous matter and does not occur if the liquid has been adequately filtered even when fine capillaries of glass (inside radius 5.59 μ) and platinum (inside radius 9.35 μ) are used.

The diameter of these capillaries, calculated from an equation depending on the viscosity and rate of flow of the liquid, agrees very closely with actual measurement and shows that if there exists any fixed layer of molecules adjacent to the walls, the thickness of such a layer does not exceed 2–3 hundredths of a micron. D. L. S.

459. The Propagation of Flame in Electric Fields. Part I. Distortion of the Flame Surface. E. M. Guénault and R. V. Wheeler. *J.C.S.*, 1931, **132**, 195.

Various attempts have been made to demonstrate the electrical nature of flame propagation by subjecting the inflaming gases to intense magnetic or electric fields. The results have been conflicting and, in those cases where positive effects have been remarked, it is not clear whether these have been due to a distortion of the flame, leading to differences in the heat loss to the confining walls, or to real differences in the reactions in, and the consequent speed of translation of, the flame front. That the shape of the flame front has a great influence on the speed of flame travel has been shown by Mason and Wheeler (*J.C.S.*, 1920, **117**, 1227).

The present paper reports experimental confirmation of the distorting effect of an electrical field on a flame surface. Mixtures of carbon monoxide and air or oxygen were ignited electrically at the centre of a glass sphere and the flame movement was recorded photographically at regular small intervals of time. An electric field could be applied between two parallel plates of a condenser consisting of platinum discs at right angles to the ignition spark gap.

The photographs show that the electric field destroys the normal symmetrical flame spread from the centre of the sphere. Up to the moment at which the flame front touches the plates and discharges the condenser, there is a movement of the flame centre towards the negative plate, due to the attraction of this plate for the positive ions. This suggests that the movement of the flame surface may be mechanical, due to the movement of the heavy positive ions dragging the flame surface with them and not necessarily due to any chemical stimulus by the electric field. It negates the view that electrons shot ahead of the flame assist its propagation. G. B. M.

Coal and Shale.

460. Oil and Coal Amalgam. Trent Process Corporation. E.P. 337,920, 3.10.29. Conv. (U.S.A.), 20.10.28.

The preparation of oil-coal amalgam is described together with the subsequent baking of this product to form briquettes. The light oil distillates from this baking are condensed and used as a starting material for the initial amalgam together with finely divided coal. Asphaltic base heavy oil is then added to form an amalgam containing 85% coal, 5–7.5% light oil and 10–7.5% heavy oil (by weight). Water also may be added before or after grinding the coal to separate the ash constituents, and the light oil can be recycled. H. C. R.

461. Low Temperature Distillation of Carbonaceous Materials. A. S. Ramage. E.P. 340,276, 17.9.29.

Carbonaceous materials are given a preliminary treatment with heated reducing gases together with mechanical agitation. By direct contact with steam the residue yields the water gas to be used in the preliminary treatment. The arrangement of the apparatus is shown and consists mainly of a mechanically rabbled shelf retort together with shaft retorts and gas producers acting on heat exchanger principles. H. C. R.

See also Abstracts Nos. 427, 428, 429.

ABSTRACTS.

Geology.

462. Can Absence of Edgewater Encroachment in Certain Oil Fields be Ascribed to Capillarity? J. Versluys. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 189-200.

This paper deals with the condition which is known to exist in some oil fields, where movement of edgewater during production is practically non-existent. This phenomena has been termed the closed type of reservoir by Herold and is said to be due to lenticularity or capillary control.

The author considers two possible conditions in a partly depleted reservoir rock: (*a*) the funicular, (*b*) the pendular. In the former, oil and generally gas, flow freely, while in the latter the gas flows freely and may be followed by oil. In a porous sediment the Jamin effect would not completely stop the flow of liquids and gas, there would be internal evaporation and recondensation in the capillary tubes. The author therefore concludes that capillary control in oilfields does not exist, and that such conditions can be explained as being due to reduced permeability in the rocks which present a greater resistance to edgewater movement. V. C. I.

463. Some Effects of Metamorphism on Certain Debris in Source Rocks. T. Stadnichenko. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 161-164.

The author describes the effect of heat on organic material. Two samples are taken, Coorongite and a deposit from Lake Balkhash, and comparisons are drawn between these and Reinschia and Pila respectively. In each case the recent deposit decomposed at a lower temperature than its fossil equivalent. The author concludes that geodynamic alteration results in raising the temperature of fusion and changing the optical characters of the original organic matter from isotropic to anisotropic. V. C. I.

464. General Geology of Eastern Texas. L. V. Decker. *Oil & Gas J.*, 1931, **29**, (43), 32.

This article gives an account of the geological factors involved in the present activity along the eastern border of the East Texas Basin.

The chief structural features are the Sabine Uplift and the East Texas basin or geosyncline. The rocks exposed along the western flank of the Uplift are all Eocene in age, and consist of the Wilcox Sand (the lowest formation) from 700 to 2000 ft. thick, and the Claiborne Group made up of the Mt. Selman at the base and the Cook Mountain at the top.

Of the subsurface stratigraphy, the Midway (about 500 ft.) lies directly below the Wilcox; this is followed by Cretaceous rocks. Though much of the detailed structural geology yet remains to be proved, it is stated that the present drilling campaign will be of great value in this connection to those geologists now studying the area.

A discussion follows on shore lines, and figures illustrating different sets of shore-line conditions are given. The author is of opinion that the sand horizons now forming along the eastern rim of the basin are not necessarily of the same age, and that each sand presents its own peculiar set of conditions. It is probable that there will be production along the east rim which is due to structure, to shore-line conditions, and to a combination of both. G. S. S.

465. Black Shale Deposit in Central New York. E. W. Hard. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 165-181.

This paper deals with the origin of the bituminous matter in the Devonian Black Shales of New York. It is concluded that these were laid down in shallow water, the source of the sediments being from the East. There were two environments, the one shallow and comparatively fresh water, the other slightly deeper and more saline. The author contends that the bituminous content of the shales is directly related to the type of decomposition rather than to the type of organic matter, and that the black shales were produced when the waters were truly saline and toxic conditions were present. V. C. I.

466. Petroleum Exploitation in the Bacau District. B. Sergescu. *Ann. Min. Roumanie*, 1931, **14**, (3), 101-106.

This article concludes the consideration of oil development in the department of Bacau by considering the remaining sections of Tetzcani, Casin, Grozesti and Harja. Active drilling in these areas has now ceased for some time though a few wells are still being pumped. The geological structure is complicated by proximity to the Nappe region of the Carpathians.

The writer condemns the idea that the oil originated in the Miocene and has migrated into the overthrust Oligocene. He considers a much more likely source to be the bituminous shales of the Oligocene, and quoted Voitesti's opinion that the source of the oil in Rumania is deeper than has been admitted hitherto. V. C. I.

Field Technology.

467. Analytical Consideration of Drilling Muds. H. H. Farnham. *Petr. Eng.*, 1931, **2** (5), 117.

Experiments have been carried out for several years upon the loss of drilling mud at high pressures into sand and sandstone formations. A variety of conclusions have been arrived at, and it is generally agreed that the functions of a drilling mud are all resultant from its colloidal nature.

Natural clays contain varying quantities and types of colloids. There are two main types. The "sol" type, which suspends itself throughout the system and, when present in high enough concentration, has the ability to suspend the larger inert mud solids which by themselves would settle out. The second type of colloid is the "gel" type. The chief characteristics of this type are that of high rate of viscosity increase with increased concentration and that of causing the liquid to gel when undisturbed.

The solids of colloidal size are mostly hydrous silica and hydrous alumina and these possess the ability to break down to colloidal dimensions in water. After colloidal size is reached, their action is dependent upon the matter in true solution in the water. A change in the Hydrogen Ion concentration (PH), which is a measure of the acidity or alkalinity of the water phase, will change sols into gels and also will destroy some gels. The solution of some neutral salts such as sodium chloride will cause some colloids to coalesce and form larger particles until the individual size is no longer within the colloidal range and the solids will settle out. Such an action as this is observed when salt or salt water is penetrated by a good drilling mud. The mud appears to curdle and free water is seen in the returns from the well.

The hydrostatic pressure produced by a column of drilling mud attempts to force the mud from the column into the formation, and since all formations are pervious to water, especially at such high pressures, there will be an

infiltration of water into the formation with a consequent deposition of solids in the walls. This will continue until (1) the resistance to further infiltration becomes too great or (2) the solids deposited in the walls of the well are sufficiently impervious to prevent flow. Actually the colloids will pass through the formation with the same ease as will water and it is in this case that the gel type colloids come into action. In contact with the wall there is no motion of fluid and the gel type colloids can then revert to the solid state and build up an impervious wall so sealing off the open hole against further loss of drilling mud.

Using Stokes formula for the rate at which a spherical particle falls through a liquid, $V=2R^2(S-S_1)g/9N$, it can be seen that as the particle size becomes smaller, where "R" is the radius, the velocity is reduced, and when the colloidal size is reached the velocity will be so low that any currents set up greater stream velocities than does the gravitational effect. The formula also shows that the velocity is inversely proportional to the viscosity of the fluid, where "N" is the viscosity of the liquid, so that by increasing this factor particles of sizes larger than colloidal may be suspended.

For fishing and casing jobs the gel forming colloids are essential to ensure a successful result.

No definite method of testing muds can be advanced until an agreement has been reached assigning definite physical characteristics to the "ideal drilling mud." This does not mean that there is only one mud for all occasions, but that for any set of conditions encountered there is always one mud that will give better results than any others. Any drilling fluid can be divided into three parts, (1) the water content, (2) the gel forming colloids, and (3) the weighting materials. By using these three divisions a means is given of evaluating any new mud in terms of other known muds.

Considering the functions of these three divisions, an ideal drilling mud may be defined as one that has the lowest density which will still provide ample factor of safety over the formation pressure and at this density has a viscosity ranging between 6 and 15 centipoises. Drilling muds conforming to the definition of "ideal drilling mud" will always contain the greatest possible reserve of gel-forming colloids which is compatible with the density and setting requirements. The driller can maintain his mud at the proper colloidal concentration by controlling the mud weight alone and by judging the proper amount of colloids to add. He will have to experiment in the reserve mud pit with the mud from the well and a definite quantity of natural clay high in gel-forming colloids. After a few round trips of the mud the driller will be able to judge whether the mud is too thin or too thick and a correction can then be made. The treatment will result in a mud weighing slightly less than the original mud, but with a viscosity exactly the same and somewhat higher in gel-forming colloids. Thus by a control of density alone the drilling mud is always assured of ample reserve of colloidal matter.

L. V. W. C.

468. Screening of Mud in Oil Well Drilling Operations. A. Grounds. *Petr. Times*, 1931, **25**, 336.

The interest shown in the preparation of rotary drilling mud has brought on to the market various admixtures and clays to make up for the deficiencies in the natural clays. The admixtures are extremely expensive and the old method of allowing the mud to run to waste has added so much to the cost of drilling wells that some method was needed to clean and conserve the mud as it left the well.

The vibrating screen has therefore made its appearance and has been so successful that the added cost of the admixtures is not now noticed in the cost of the well.

The screen consists of a steel frame in which the meshes are mounted so that there is no obstruction to the material down the deck. The body is carried on two pipe stringers held at their ends in flat coil springs. Bolted to the body is the shaft carrying the steel pulley which runs at 1,800 r.p.m. and which is thrown out of balance by a series of small weights. This has the effect of giving the screen an elliptical motion with a frequency of 1,800 per minute and an amplitude of about ¼ in. A 30-mesh cloth is supported by a ½ in. metal mesh.

The angle at which the screen is set is about 12°, and this angularity has been found suitable in most cases.

The fluid from the well is delivered along the top deck of the screen, the fluid passes through the screen cloth into a trench leading to the pump sump, whilst the course rejects fall off the lower end of the screen into a reject pit in which a steam jet is placed to get out the cuttings to a larger outside dump. L. V. W. C.

469. Prevention and Control of Oil Field Fires. W. C. Lynch. *Petr. World*, 1931, **28**, 71.

The extinguishing of an oil well fire may well cause greater damage than the fire itself if the flow from the well is allowed to blow freely over the district. For this reason, in congested locations, a pilot jet is often placed at the well mouth to ensure the firing of the well in the event of its sanding up and then breaking loose again. Tunnelling and the use of hoods and valves have been used to handle the flows from these wells without first extinguishing the fire.

For extinguishing oil pools, sumps, tanks, etc., foam is the safest and best medium to use, in most cases, but the method of application should be thoroughly understood. The foam must be applied gently if progress in extinction is to be rapid. Owing to the fact that crude oils of varying gasoline content may be encountered in the same field, consideration must be given to the medium to be used. In the crude oil fresh from the well we find a higher gasoline content than in the oil which has been allowed to stand in sumps for an appreciable period. The light crudes burn freely, are easily ignited and difficult to extinguish, while the heavy sump oils do not ignite so readily and, although they burn freely, once they have caught properly, they are fairly easy to extinguish providing the gasoline content is low.

For these heavier oils a sprinkle of water will often extinguish the fire, but the spray must be very fine if successful results are to be achieved and special nozzles are necessary for suitable sprays.

From a number of experiments made in the extinction of oil fires it is definitely established that water has a place in oil fire work. The water, however, must be used intelligently. It is essential that a fire spray should be used for short periods only if a "boil-over" is to be prevented. This "boil-over" results from the use of too great a quantity of water. The hot layer of oil formed on the surface deepens until it reaches the water under the oil. This water evaporates with such rapidity that the oil is forced out.

For the lighter oils foam is the more suitable medium. Here, again, great care should be exercised in the quantity used. A "boil-over" easily results from the use of too large a quantity of foam.

The spray, of whatever medium is used, should be as fine as possible and the conventional round nozzle should not be used.

In the case of fires due to a breakage in pipe-lines, or pipe-line apparatus, the pressure should be relieved in some manner and then streams of foam and mud, water or steam should be forcibly applied to the break in order to make a gap in the flow. L. V. W. C.

470. Selection of Oil Well Casing Based on California Practice. J. L. Gignoux. *J. Amer. Petr. Inst.*, 1930, **11** (75), 4–14.

The design of casing programmes is of considerable importance to the operator because of the cost, which is 23 to 35% of the entire initial cost of the well. Unless the proper-sized strings are successfully landed at the desired depths additional strings, not originally contemplated, will be required, resulting in loss of diameter and increased expenditure.

The results of a study of casing practices on about 225 wells representative of both shallow and deep operations now being carried out in California, are shown.

Since a theoretical analysis appeared to have its limitations the problem was approached from an empirical consideration. The clearance ratio, that is the relationships between the hole clearance and the diameter of the open hole expressed as a ratio, $D_1—D_2/2D_1$, where D_1 is open hole diam., and D_2 outside diam. of pipe couplings, for strings upon which records were obtained were calculated and the open hole or friction in which they were set was determined. The averages of these clearance ratios have been plotted against the friction and the graphs are given. It can be seen from these graphs that the clearance ratios are greater for the various groups of casing in the larger sizes, and that all ratios increase with greater friction depths. This also indicates an increase in hole clearance for larger casing sizes and increased friction lengths. A table is supplied showing the relationships between the various groups of casing sizes and the maximum and minimum of the average clearance ratios for average friction lengths.

Programmes requiring under-reaming are compared with those in which the practice is eliminated. Although under-reaming may reduce the cost of a well there are two disadvantages which are difficult to evaluate. It not only increases drilling time and adds an operation which may possibly result in a fishing job, but it penalises the obtaining of early production, a serious matter in a competitive area. The practice of under-reaming is rapidly decreasing and is rarely used except in an emergency. It has been suggested that under-reaming to remove the drilling mud from the walls of wells in fields where the static head of fluid is low will enable the fluid to enter much more freely.

The safety factor practices in tensile stress of casing joints and collapsing stress of casing are shown. Though these appear to be justified by common usage, there are many indeterminable factors entering into the problem tending to make it advisable to increase the wall thickness of the selected casing, to the utmost limit consistent with a reasonably economical policy.

Greater care should be taken to see that the holes are in the proper condition before casing is run, and the clearance ratios well established by use will not apply in cases where the drilling muds are not of good quality, or the reaming or under-reaming operations are not thoroughly carried out.

Casing lists cannot be increased or decreased until a uniform method of designing programmes has been established. L. V. W. C.

471. Sand Cutting Reduced by Flow Velocity Control. W. L. Baker. *Petr. Eng.*, **2** (5), 92, 93.

Reduction of flow velocity by the use of chokes and expansion chambers, and the elimination of bends in flow lines by the adaption of straight lines is being used in a number of hook ups.

Two separators placed on opposite sides of the derrick are connected by straight flow lines from a cushion drum placed over the well mouth connections, thus obviating angles or bends against which the sand in the oil may

impinge. The only change in direction of flow the oil makes is in the drum, where it changes from vertical to horizontal flow.

A reduction in velocity of flow is secured at the bottom of the cushion drum by means of a cast steel choke 4 ft. long by 2in. or 3in. diam. The drum acts as an expansion chamber and the flow lines of 12¼in. diam. pipe increase the expansion area. The fluid from the well flows through the choke to the drum and thence through the flow lines to the separators, where the mud and sand settle to the bottom. The separators are cone-shaped, each with an 8in. discharge line in the centre for carrying the mud and sand to the slush pits.

When the well has cleaned itself the outlet line for the mud is reduced in diameter for removing the sand. The gas comes out of the separator at the top, and the oil is taken off at the side.

All the parts of this equipment are specially manufactured to withstand the heavy duties they have to perform. L. V. W. C.

472. Method of Repressuring Individual Wells. N. Williams. *Oil and Gas J.*, 1931, **29** (42), 38.

With a view to increasing producing efficiency and conserving natural resources the Gulf Production Co. have commenced a repressuring programme on their Hankamer Field. The work so far has been experimental, but it has resulted in material reductions in gas-oil ratio, increases in production and maintenance of flowing life of wells.

It has been found impossible to institute a general repressuring or pressure maintenance programme owing to the fact that the eleven wells completed represent what are believed to be seven distinct producing zones. It is estimated that not more than three wells are producing from any one of the horizons.

Two of the eleven wells completed have had relatively high pressures, and, producing only a small quantity of oil, each has had an excessive gas-oil ratio. The remainder of the wells, on the other hand, have had very little pressure, and after a short life need some artificial means to maintain production.

The gas energy from the two high pressure wells has been used in what might be termed a natural gas lift. The gas is injected into the lift wells at the pressure that exists at the two gas wells, thus eliminating the necessity for compressors.

The gas from one of the high-pressure wells at a pressure of 825lbs. is being injected into one well. The gas from the second high-pressure well is being injected into two near-by wells at a pressure of 900lbs.

In the first case the gas is being injected through tubing and out of the casing, whilst in the second case the order is reversed.

At present no separators are in use, but plans are being made for the gas to pass through separators to trap whatever oil there may be, and to provide a gas as nearly dry as possible for injection purposes.

In the operation of the lift system good results have been attained, and the gas that was previously wasted is now being conserved. In addition, the gas-oil ratio has been reduced considerably.

Pumping has been completely eliminated, and the lifting costs have been considerably reduced. L. V. W. C.

473. Shutting off Intermediate Water at Santa Fé Springs. B. Mills. *Oil Weekly*, 1931, **60** (12), 25.

The system used for shutting off intermediate water at Santa Fé Springs is devoid of elaborate technique but has proved effective because the trouble has been overcome only after the determination of the level from which the water was entering the hole.

So high had the percentage of water become that wells have ceased to be commercial producers. Thin water sands found just below the shoe of the final water string have been a source of constant worry on these fields.

In pumping wells, the encroachment of sand will usually continue until the hole is filled to some level which is able to yield sufficient force to hold the sand in check.

It has been found that pumping wells which are bothered by intermediate water will gradually sand up to exactly the point at which the water is coming into the hole. This sand forms a solid plug and the well produces only from the zone above the streak of water sand and gets the full effect of the intermediate flow of water. If the streak of intermediate sand is near the shoe the production will fall considerably.

Careful observation showed that the sand stopped packing in the hole at a certain level and that this level changed very little. The conclusion was that the water was entering the hole at this point.

Running the bailer revealed that the sand was well packed and would furnish an excellent support for the operations.

Cement was dumped in by a dumping bailer until the formation would take no more cement. The first few times the bailer was dumped there was no rise in the level of the sand and cement, proving that the cement was going into the formation. When the formation would take no more cement the bailer was run in and cement dumped to raise the cement to a safe position above the water stratum. The hole was then cleaned to bottom and put on the pump again. The percentage of water was then found to be considerably lower showing that the dumping of cement with the bailer had plugged off the intermediate water sand. L. V. W. C.

474. Study of the Production Curves for the 8,500-foot Horizon, Big Lake Oilfield, Reagon Co., Texas. K. S. Ritchie. *Petr. Div., Amer. Inst. Min. Met. Eng.*, 0.2.31.

The first well to produce oil from a depth of 8,500 ft. was University 1-B of Group No. One Oil Co., and for over 17 months was the only producer from this horizon. The only change at the surface to regulate flow was the application of 185 lbs. back pressure after several months. The daily production increased from 40 brls. to 2,926 brls. and, after six months of a decline period, then set its record of 2,950 brls.

Cable drilling was used and work commenced on February 8, 1926. Oil and gas were obtained at 2,469 ft. and in August, after unsatisfactory production from the Texon Pay at 3,010 ft., further deepening was started. On December 1st, 1928, oil and gas was found at 8,523 ft. The last casing set was 6,176 ft. of 5-$\frac{1}{2}$ in.

The production curve is unusual, being parabolic instead of hyperbolic. Gas production followed the trend of oil production until other wells began producing, when it started to decline, although oil yield started to increase again. After declining in the first six months, the gas-oil ratio remained fairly constant and declined further when the oil production started to rise again. G. S.

475. Use of the Cable Tool Core Barrel in Recovering Samples of Oil Sands. P. D. Torrey. *Pet. Eng.*, 1931, **2** (5), 106.

The cable tool core barrel has been extensively used in the Appalachian oilfields for obtaining cores from oil sands, and this method has been found not only most effective and practicable but also reasonable in cost.

One common type of core barrel used consists of two main parts, an outer drilling barrel and an inner core retaining tube, and both these parts are made of heavy seamless tubing. The drill barrel is composed of the drill

barrel head, the main drill barrel and the drill shoe and is fitted with the normal type of box and pin. Holes drilled in the side allow water circulation through a chamber at the lower end, which is closed by a back pressure valve. The drill barrel is a cylinder of seamless steel and is screwed on to the drill barrel head and the drill shoe, which is slightly expanded at its lower end and is equipped with stellited teeth, is screwed to the lower end of the drill barrel. The inner diameter of the shoe is smaller than the inner diameter of the drill barrel so that a collar is formed which prevents the inner core tube from falling out while the tools are in the hole. Water courses on the inner part of the drill shoe allow free water circulation between drill barrel and core tube.

The core tube is composed of three parts: core tube head, main core tube and the trimmer shoe. The core tube head is a short piece of tubing expanded on the outside at the top to hold the core tube in the drill barrel and is fitted with a ball check valve at the bottom to allow the escape of water as the core tube fills with core. The core tube head is screwed to the top of the main core tube and the trimmer shoe is screwed on the lower end. The shoe is slightly bevelled and its cutting edge is faced with stellite.

The operation of coring is similar to ordinary drilling operations with cable tools. The string of tools is as usual except in hard sand when as heavy a sinker bar as possible should be used.

The outer drill barrel is first screwed to the stem and then the barrel is allowed to drop over the inner core tube. The drill shoe is then screwed on. In this condition the inner core tube hangs down below the drill barrel being held by the expanded head of the core tube resting on the shoulder above the drill shoe.

The hole should be cleaned out before running the core barrel and enough water run in to cover the barrel on the up stroke.

A slow motion should be imparted to the walking beam, about 33 strokes per min. gives satisfactory results under most conditions.

Whilst coring is in operation the inner core tube does not leave the bottom of the hole, the drill barrel cutting out a cylinder of the formation which is pushed into the core tube and held there by the retaining spring set in the trimmer shoe. On the up stroke the water valve in the drill barrel head opens, drawing water into the space between the inner core tube and the drill barrel and on the down stroke this valve and the valve in the head of the inner core tube closes, forcing the water out around the teeth of the drill shoe and prevents accumulation of cuttings in the bottom of the hole.

After the temper screw has been run out the core barrel is pulled from the hole, and by removing the drill shoe the core can be extracted. It is advisable to use a new drill shoe for each run to ensure the gauge of the hole.

Under ordinary conditions progress with core barrel is as rapid as with standard tools but time is lost in the changing of the tools. L. V. W. C.

476. Practical Interpretation of Core Analysis. L. S. Panyity. *Petr. Div., Amer. Inst. Min. Met. Eng.*, 0.2.1931.

The value of screen analysis of sand is stressed, particularly for fields in which repressuring is used. The usual type of "core-chart" is used, but grain size is represented by percentages passing through various sieves. Thus only one line is necessary to represent porosity, saturation and grain-size of any sample.

It is suggested that the expression of oil content as a percentage of total porosity be eliminated or replaced by brls./acre-foot of sand.

Grain-size determines to a large extent the permeability of the rock, but cementing material is also a large factor as it passes through the 200-mesh screen and this size has a great bearing on permeability. G. S.

477. New Features in Pipe-line Station. L. E. Smith. *Inter. Petr. Tech.*, 1931, **8** (3), 14.

Spray ponds and louvre towers for cooling engine waters are entirely eliminated from the pump stations on the Gulf Pipe Line Company's system from new Perryman to Buckeye Junction.

A heat exchanger has been adapted for cooling the engine water. Return water from the engine jackets enters the heat exchanger and is cooled by the fresh oil from the storage tanks, prior to its entering the pumps for dispatch into the pipe-line. The amount of heat permitted to enter the heater, or cooler as it has now become, controls the temperature. The correct amount of water to keep the temperature at the desired point is admitted by manually operated cocks. The system is designed to cool 175 gals./min. of fresh water from 110° F. down to 95° F. by the circulation through the cooler of 540 gals./min. of 39° A.P.I. gravity oil, which enter the cooler tubes at 90° F. and leave at a maximum of 99° F. The water system is entirely enclosed, the water travelling from the engine through the cooler and back to the engine again. Except in extremely hot weather the loss in water is estimated at not more than one gallon in 24 hours. This water economy and the ease with which water is handled in winter justify this internal cooling system.

The water supply will be from rainfall collected in a 250 brl. capacity cistern.

The cooler consists of a 12 in. diam. shell fitted with Victaulic couplings at either end with a header. The 16 ft. long chamber is fitted with 104 ¾ in. tubes running lengthwise. Communication between the two headers is through these tubes and only oil passes through them. The oil is forced through the tubes by a centrifugal pump capable of handling 600 gals./min., and is then fed to the main pump in the line. The water flows through the main shell in the reverse direction to the oil thus bringing the hottest water into contact with the warmest oil end of the cooler first.

Blower heaters are used for heating the station. A radiator is placed in the shell of the heater and steam is circulated at about 4 lbs. pressure through the top. A 12-blade fan blows through the radiator, causing a stream of warm air to pass into the room and this process results in the condensation of the steam and the water formed passes out through the bottom and is returned to the boiler.

Two lubricating oil purifying centrifuges each serve two Diesel engines. Lubricating oil for these engines is contained in tanks sunk beneath the floor and the oil is pumped from these tanks to all the main engine bearings at a pressure of 14 lbs. The circulating oil is periodically switched to sump tanks for cleaning purposes.

Another feature of the station is the manner in which the engine exhaust is muffled. An 8 in. exhaust pipe, passing through a 10 in. water cooling jacket, carries the exhaust gases of each engine to a foot muffler on the outside of the building. The noise usually associated with such large engines is entirely eliminated.

A number of innovations were introduced into the making of the line manifold. Wherever possible welded joints were used and tube turns were used in place of ells at every opportunity.

In tanks handling sour crudes inside aluminium foil was used at the coating of the top ring thus preventing a certain amount of corrosion at the point most affected by the sulphur oils. L. V. W. C.

478. Concrete Storage Must be Impervious. J. F. Hough. *Oil & Gas J.*, 1931, **29** (37), 37.

The lasting quality of a concrete oil reservoir depends as much on its resistance to disintegration by water or salts in solution, as on its structural strength. The water-cement ratio of the mix must be adjusted to give the necessary structural strength to the concrete, whereas, if the mix has been regulated to give a resulting liquid tight surface, it is probable that the strength requirements will also be amply met. Design of the cement to give the necessary durability is often, therefore, more important than design to give strength.

Disintegration may be due to chemical action, dissolving out the ingredients of the cement, or to physical action by the freezing of penetrated water or the crystallisation of salts after evaporation from solutions that have penetrated into the concrete.

When designing a cement mix, full consideration must be paid to the necessary amount of water and to the use of a combination of aggregate which will give a plastic workable mix and remain homogeneous during placing, setting and curing. Besides the necessary amount of water to complete the chemical reaction, further water is necessary to render the cement plastic enough to handle, while excess of water will reduce its strength and durability.

Once the mix has been decided upon, favourable conditions for the setting and curing of the cement must be provided. The chemical reactions involved in the setting of cement require (1) time, (2) favourable temperature, (3) the combined presence of water to give to the final concrete its necessary strength and liquid tightness. After the cement has set, hardening takes place as the material dries, this proceeding for an indefinite time, progress being rapid for the first ten days or a fortnight, during which the presence of moisture is essential to prevent the ceasing of the chemical action between the cement and the water. Curing should be continued for at least ten or fourteen days longer if possible.

A table is given to enable an estimation to be made of the amount of water necessary for concrete made from different aggregates, but it is pointed out that a trial batch should first be made, before the final mix is decided upon. The precautions necessary during mixing, handling and placing are described with some hints on the avoidance of segregation of components of the aggregate, etc. C. L. G.

479. Deep Drilling Apparatus. W. R. Macdonald. E.P. 328,692, 30.4.30.

This invention relates to the method of drilling by means of a high speed percussive tool constructed so that the frequency of the blows on the bit can be controlled from the surface. An effort has been made to increase the frequency of blows to approach the natural frequency of the stratum being drilled. The frequency in this case is between fifty and ninety periods per second.

The apparatus may be operated by compressed gas or liquid under pressure or a combination of both. The action is similar to that of the ordinary hammer drill adapted for use in the deep holes. A piston, operated by the air or liquid under pressure, moves freely and strikes the tool at each reciprocation. L. V. W. C.

480. Rotary Drill Bit. L. C. Black. U.S.P. 1,791,186, 3.2.31. Appl., 8.3.23.

This rotary bit consists of a twisted blade with cutting edges. The centre longitudinal portion is equipped with a passage and the upper end has a dovetail slot which fits in a shank provided with a water passage which coincides with the passage on the blade member. A collar on the shank engages the upper end of the blade member. L. V. W. C.

481. Gas Lift Pump. J. H. Slocum. U.S.P. 1,791,513, 10.2.31. Appl., 3.7.29.

This pump consists of a hollow body in which is formed a high-pressure chamber, for receiving the high-pressure gas, with an outlet on the upper side of the body having an eccentric tapered socket. A down pipe with a tapered tip runs from the surface to the pump body for conveying the high-pressure gas and this tip is capable of seating in the eccentric tapered socket. A flow pipe is arranged around the down pipe, in such a way that the down pipe lies aginst the inner wall of the flow pipe, and is connected to the body of the pump. A nozzle communicates with the high-pressure chamber for delivering the high-pressure gas to the lower end of the flow pipe and a means is supplied, in association with the nozzle, for retaining the pumped liquid.
L. V. W. C.

482. Apparatus for Flowing Wells by Air Lift. W. L. Martin. U.S.P. 1,792,107, 10.2.31. Appl., 1.2.28.

A packer is set in the casing and tubing run through the packer. A short conduit in the tubing communicates with the casing both above and below the packer and a cap is placed on the lower end of the conduit which has a distributing chamber and ducts leading from the chamber to communicate with the casing below the packer. Nipples in the tubing communicate with the casing above the packer and air under pressure is delivered to the casing above the packer.
L. V. W. C.

483. Reamer. J. W. MacClatchie. U.S.P. 1,792,697, 17.2.31. Appl., 4.4.28.

This reamer consists of a hollow steel body equipped with a bushing with a cutter assembly mounted in it and with means for radially adjusting the bushing to compensate for wear.
L. V. W. C.

484. Reamer for Earth Boring Apparatus. C. E. Reed. U.S.P. 1,792,869, 17.2.31 Appl., 21.3.27.

A side recess is cut in the reamer body, and auxiliary recesses are cut at the top and bottom of the body. A reamer roller-bearing sleeve, with an enlargement at its upper end and a removable collar on the sleeve at its lower end, bear on the walls of the auxiliary recesses to take the lateral thrust from the reamer roller and a pin passes through the sleeve and supports it in the body.
L. V. W. C.

485. Expansive Rotary Under-reamer. J. T. Barkelew. U.S.P. 1,793,988. 24.2.31 Appl., 19.11.29.

In this under-reamer, cutters, mounted in the main body, are permitted outward and inward motion for expansion and contraction. One of the cutters is movable longitudinally with respect to the other. The cutters are in contact with each other inside the body, and by means of inclined surfaces any downward movement of the longitudinally moving cutter causes both elements to move in an outward direction. Upward movement causes both the elements to move in an inward direction due to the inclined surfaces at the lower end of the cutters.
L. V. W. C.

486. Overshot for Well Fishing. E. W. Forrow. U.S.P. 1,794,226, 24.2.31. Appl., 20.3.28.

This overshot varies from the general type of overshot in that it has a means for releasing the tool from the object picked up. It is provided with a latch, and by a longitudinal movement between the latch and the body of the tool the object can be released.
L. V. W. C.

487. Apparatus for Pumping Wells. A. F. White. U.S.P. 1,794,427, 3.3.31 Appl., 6.8.28.

Into the pump chamber of this displacement pump compressed gas is fed through a contracting valve. This control valve consists of an intake passage communicating with a compressed gas supply, a valve chamber connected to the intake passage and communicating with the pump chamber. At the junction of the intake passage and valve chamber a valve seat is provided, and a piston is formed in the intake passage by a gas inlet valve and stem. A notch is cut in this stem, so arranged that compressed gas will flow past the gas inlet valve when this valve has moved a predetermined distance. Into the head of the valve, gas passages are cut upon the periphery. In addition a moving plunger is placed in the valve chamber having gas passages on its periphery, and a spring operates between the gas inlet valve and the plunger. Between the pumping chamber and the exterior of the displacement pump a number of gas vents are provided having valve seats. A valve member carries the vent valves and is engaged with the plunger. Displacement of the plunger closes the vents. A second spring is provided to open the vents until the compressed gas pressure exceeds a predetermined value and displaces the inlet valve and plunger. L. V. W. C.

488. Hydraulic Trip Spear. F. Stone. U.S.P. 1,794,652. 3.3.31. Appl., 25.5.29.

This trip spear consists of a steel body with a sleeve so mounted that it can reciprocate freely over the main member. An opening is cut through the wall of the sleeve to enable a gripping device to operate through it. This gripping device moves radially into and out of working position, and is directly carried by the main body. The reciprocation of the sleeve moves the gripping jaw from the work to the out of work position. L. V. W. C.

489. Rotary Expanding Under-reamer. J. J. Santiago. U.S.P. 1,794,735, 3.3.31. Appl., 22.10.27.

The main parts of this expanding under-reamer are a tubular steel body, a mandrel and a movable cutter. The mandrel has bearing faces on the inner surface of the steel body immediately opposite the bearing surface of the cutter against the mandrel. The cutter bearing against the mandrel is give an rotary movement by the rotation of the mandrel. L. V. W. C.

See also Abstract No. 603.

Crude Oil.

490. Refining Five South Texas Crudes. G. Egloff, E. F. Nelson, P. Truesdell. *Oil and Gas J.*, 1931, **29** (40), 30.

The Refugio field in S. Texas yields five types of crude oil which vary considerably in properties, according to the depth of the producing level from which they are obtained. The 6400-ft. sand yields a crude oil of sulphur content 0·021% which contains 91% of gasoline (F.B.P. 424° F.), 5·72% kerosine and 3·3% bottoms and loss, and the gasoline and kerosine need only a caustic wash to remove H_2S in order to produce a marketable

product. The 5500-ft. sand yields three types of crude having the following properties :—

	Type 1.	Type 2.	Type 3.
A.P.I. gravity	29·5°	37·0°	38·3°
Sulphur	0·16%	0·13%	0·13%
400° F.B.P. gasoline	28·68%	48·0%	44·9%
437° ,, ,, ...	—	54·1%	51·2%
Octane No. of gasoline	—	57	54

The crude from 3700-ft. sand is a natural cracking stock, naphthenic in character, having the following properties: A.P.I. grav., 23·2°; I.B.P., 388° F.; F.B.P., 754° F.; sulphur, 0·2%; flash pt. (Cleveland), 185° F.; cold test, below 0° F.; viscosity, Saybolt Furol at 77° F., 14 secs.

This oil was subjected to cracking under a pressure of 350 lb. and at temperatures varying from 920° to 950° F. using the "flashing process." Five tests were made, yielding 59·5/61·2% of gasoline, 24·3/37·0% of residue and 407/685 cub. ft. gas (containing over 2 gal. of gasoline per 1000 cub. ft.) per brl. The properties of the gasolines were: A.P.I. grav., 44·6/46·0°; I.B.P., 94/101° F.; 20% distilling over at 210/230° F.; F.B.P., 413/438° F.; octane No., 88/90 (Ethyl standard = 74).

The properties of the "flashed" residues obtained were: A.P.I. grav., 1·1/8·6°; B. S. & W. content, trace/0·3%; Viscosity Saybolt Furol at 122° F., 31/240 sec.; flash pt. (Cleveland), 225°/250° F.; cold test, 2°/38° F.; distilling to 572° F., 21/26%.

The pressure distillate is easily refined, the method adopted being: (1) Agitation with acid (2 lb./brl.), sludge settled and drawn off; (2) water wash; (3) neutralisation with 1% by vol. of 16° Bé sodium plumbite solution, settling and removal of sludge; (4) distillation with fire and steam at a low temperature; (5) freshly distilled gasoline treated with 1% by vol. of 2° Bé caustic solution. The chemical compositions of straight run and cracked Refugio gasolines are :—

	Straight Run.	Cracked.
Unsaturates	2·2%	30·0%
Aromatics	8·7%	32·8%
Naphthenes	23·1%	25·0%
Paraffins	66·0%	12·2%

Topped residues from the three 5500-ft. sand crudes were cracked under a pressure of 200/300 lb. at temperatures ranging from 832° to 840° F., yielding 47·5/52% of gasoline (F.B.P., 435/439° F.; Knock rating, 75/79, Octane No.) and 16·2/17·9% gas oil. The yields of gas and coke per brl. of cracking stock were 962/1046 cub. ft. and 45/56 lb. respectively. The pressure distillate was chemically treated and distilled with steam to obtain the gasoline as distillate and the gas oil as bottoms. The gross yields from the three crudes were thus: Str. run gasoline, 27·7/54·1%; cracked gasoline, 34·2/23·4%; Diesel or furnace oil, 12·1/7·2%; coke, gas and loss, 26·0/15·3%.

R. A. E.

491. TREATMENT OF OILS. Petroleum Conversion Corporation. E.P. 341,925, 23.1.31. Appl., 23.10.30. (Conv. U.S.A., 6.11.28.)

The invention relates to a method and apparatus for distilling oils, and in particular lubricating oils from suitable crudes. The process consists in introducing a large quantity of a hot inert or neutral gas into a quantity of oil undergoing distillation. The quantities are adjusted so that the evaporation of the oils occurs without the use of a substantial degree of vacuum. A continuously operated plant is described which embodies arrangements which permit both ingoing gas and oil to be brought into heat exchange relation with outgoing gas and oil respectively. The equipment is arranged for continuous distillation so that the heat carrier gas is caused to bubble through the body of the oil, while the cool oil feed is showered through the gas space above the liquid level in the distillation chamber. R. W. L. C.

492. Fractional Extraction of Mineral Oils. T. W. Ferris, Assr. to Atlantic Refinery Co. U.S.P. 1,788,569, 13.1.31. Appl., 16.7.27.

The invention relates to the separation of crude petroleum into fractions of different chemical compositions, but of approximately the same distillation range. The process, which is applicable to viscous oils containing both paraffinic and naphthenic series of hydrocarbons, consists in extracting the oil with a nitro-derivative of an aromatic hydrocarbon, more particularly nitrobenzene. This treatment effects the solution of a portion of the oil richer in naphthenic hydrocarbons, while the undissolved portion is correspondingly richer in paraffinic hydrocarbons.

The preferred method of carrying out the process consists in agitating the oil with nitrobenzene in the proportions of one volume of oil and two volumes of nitro-body and raising the temperature of the mixture to about 60° C. to effect complete solution. The mixture is then cooled to about 10° C., when a two-layer system is formed and separation of the upper and lower layers made. The treatment may be repeated with the production of nitrobenzene extracts which are somewhat less naphthenic than the first extract. The nitrobenzene is removed from both the upper and lower layers by distillation. R. W. L. C.

493. Treating Crude Oil having Tar and Asphalt Content. W. S. Baylis, Assr. to Filtrol Co. U.S.P. 1,788,653, 13.1.31. Appl., 12.11.27.

The invention provides a process by means of which lubricating oils can be distilled, or fractionated, and purified at the same time. Acid treatment with the accompanying sludge losses is obviated. The process consists in vaporising the lighter oil fractions from a crude oil so that the asphalt remains in the distillation vessel. The lubricating cut is fractionally condensed in a bubble tower from the base of which it flows to an agitator where it is mixed with an adsorptive clay. The adsorptive clay is usually one which has been activated with acid and is free from sulphate salts, but may carry a small quantity of sulphuric acid on its surfaces. The mixture of lubricating oil cut and clay is raised, first by heat exchange and then by means of a pipe still, to a temperature sufficient to vaporise the lighter fractions. The mixture is then discharged at about 750° F. into an atmospheric fractionating column. The lighter lubricating oil fractions are vaporised and taken off in the usual manner. The mixture of unvaporised residue or cylinder stock and clay is withdrawn from the base of the tower and passed through a cooler. At this point a suitable solvent is introduced to enable the residue to be handled more readily in the filtering equipment employed to remove the clay from the cylinder stock. R. W. L. C.

494. Treating Crude Oil of the Pennsylvania Type. W. S. Baylis. Assr. to Filtrol Co. U.S.P. 1,788,654, 13.1.31. Appl., 12.11.27.

The process described in this invention is particularly applicable to the treatment of crude oils such as paraffin base Pennsylvania oil which is free from tar or asphalt. A suitable crude oil is fed in a continuous supply to an agitator, where it is mixed with a suitable adsorptive agent, which is preferably the acid-treated clay known as "Filtrol." The mixture is then passed through heat exchangers to a pipe still in which it is raised to a temperature of about 750° F. The hot mixture is discharged into the vaporising section of a fractionating tower. The lighter fractions comprising wax distillates, gas oil and mineral seal oil condense in the order of decreasing boiling points and are taken off in the usual manner. The cylinder stock collects in the lower end of the column, from which it is withdrawn together with the suspended clay. It is cooled and filtered from

the clay after mixing with a suitable solvent to facilitate filtration. The recovered clay may be reactivated and used again in the process. In the case of the use of "Filtrol" the activated material may be made to carry a small proportion of free acid on its surfaces with advantageous results.

R. W. L. C.

495. Separating Emulsions. F. R. Moser, Assr. to N.V. de Bataafsche Petr. Mij. U.S.P. 1,788,684, 13.1.31. Appl., 3.9.24.

Petroleum-water emulsions are treated with a colloidal material obtained by treating mineral lubricating oil (substantially free from substances liable to resinify) with strong sulphuric acid in excess. The sludge so formed is diluted with water and the unaltered oil removed, whereby an aqueous liquid containing colloidal matter results. W. H. T.

See also Abstract No. 563.

Natural Gas.

496. Natural Gas Leakage from Pipelines. *Oil and Gas J.*, 1931, **29** (41), 23.

Bureau of Mines Bulletin 265 (Leakage from Natural Gas Transmission Lines) describes a satisfactory method for determining the rate of leakage from pipelines and gives characteristic curves of leakage against pressure. Means of locating leaks are given and the necessity of keeping adequate records of leakage and of taking greater precautions in constructing lines are emphasised.

Since the publication of the above bulletin the subject has been closely studied by operating companies and many improvements instituted. (Copious references to papers given.)

The most satisfactory method of estimating the loss by leakage is to shut off the particular section of line, preferably with blind discs between flanges or with bull plugs if the valves are not absolutely leak-proof, and to measure the drop in pressure over a definite time. The time interval should not extend over three hours to avoid variations in temperature. The rate of leakage is usually recorded in terms of the number of cubic feet lost per year per mile of 3 in. line at a gauge pressure of 100 lb./sq. in.

When it is desired to estimate the leakage from a main trunk line which cannot be blanked off, this is determined by measuring the pressure at every place where gas enters or leaves the line. This test naturally requires great accuracy in the measuring instruments.

The rate of leakage usually varies inversely as the pressure for pressures between 300 and 600 lbs./sq. in. For low rates of leakage, however, the relation approaches an equation based on the square of the gauge pressure. An investigation of the leakage losses in a line will draw attention to the possibility of an unnecessarily high pressure being used and to the presence of badly plugged outlet connections, poor screw fittings, etc. There is, however, a minimum leakage factor beyond which economic considerations do not justify repair of the line. C. L. G.

497. What Determines the Value of Absorption Oil. F. L. Kallam. *Chem. Met. Eng.*, 1931, **38** (2), 78-81.

Twenty-four samples of absorption oil, as used for the extraction of gasoline from natural gas have been obtained from refineries situated in various parts of the U.S.A., so that the average properties may be considered as represen-

tative of the average material used. The following properties of these samples and of 34 samples of used oil from absorption plants throughout the country have been examined :—(1) A.P.I. gravity @ 60° F., (2) viscosities Saybolt over a range of temperature from 70° to 200° F., (3) an average boiling point obtained by plotting graphically the boiling-point curve of 100 c.c. of the sample distilled in a 500 c.c. Hempel flask using jack-chain in the column to produce a reflux action, (4) saturation of used oils as measured by percentage distilling over at a top column temperature of 300° F., (5) mol. wts. by the cryolitic method, (6) demulsibility at temperatures varying from 70° to 200° F. by a special method using 40 c.c. of distilled water, 20 c.c. of oil, agitating violently for 3 min. and measuring the time required for the oil and water to separate, (7) vapour pressure curves over a range of temperature 70° to 300° F. using a method enabling extrapolation to zero vapour space. The results are shown in a series of charts giving maximum, minimum and, where possible, average values for both unused and used oils.

The curves clearly demonstrate that used oils as compared with unused are characterised by :—(1) A marked change in distillation curve, lower I.B.P., high F.B.P., higher average b.pt., lower distillation recovery and increased residuum content, (2) lower A.P.I. gravity, (3) increased mol. wt. for similar average b.pt., (4) increased viscosity, (5) higher vapour pressures at temperatures below 210° F. and generally lower vapour pressures at higher temperatures, (6) emulsifying tendency is appreciably greater over the whole range of temperature. For individual oils it decreases with rise in temperature. Generally the emulsifying tendency at 200° F. is greater as the residue on distillation increases, and, given equal residuum content, the used oils have the higher tendency.

It is also shown that the "saturation" figure, by which the stripping efficiency is now usually judged, bears no relation to vapour pressure and is therefore considered to be unreliable. As the determination of residual gasoline in the absorbent is important, an effort has been made to relate vapour pressure to other properties. Neither I.B.P. nor A.P.I. gravity are satisfactory, but vapour pressure at 300° F. plotted against the average b.pt. shows the latter to give a reasonable guide. Similarly both A.P.I. gravity and average b.pt. give a rough guide to mol. wt.

From the data presented it is recommended that in selecting a new oil the following points should be taken into account :—(1) Alteration in properties during use. (2) Oil of the lowest practicable mol. wt. should be used. This is generally 200 for plants of older type, but new plants should be designed to use oil of 150 mol. wt. (3) The average b.pt. and max. F.B.P. should be specified to give a reasonable control over vapour pressure and gravity, I.B.P. and gravity can then be omitted. (4) A max. distillation residue or demulsibility factor should be specified. (5) The cloud test of the oil should be below the lowest operating temperature to prevent solidification of the oil and hindrance to separation of emulsified water. (6) Max. viscosity requirements at a series of temperatures covering the range of operation should be specified. Viscosity tests are useful in operation as an indirect check on content of gum and oxidised products in the oil, which decrease efficiency. R. A. E.

498. Some Methods Concerning Gasoline Plant Design. W. F. Cerini. *Petr. Eng.*, 1931, **2** (5), 84.

In the design of a natural gas absorption plant, the quality and quantity of the liquid and vapours resulting from absorption, compression, condensation, heating, venting and rectification, and the optimum rate of circulation

of the lean oil can be determined from a consideration of the composition of the natural gas. There are two formulæ which may be used in this connection, (1) the Kremser Theory (A. Kremser, *Proc. Calif. Nat. Gas Assoc.*, 1930, 5 (2)), and (2) the Murray formula (W. Murray, *Ind. Eng. Chem.*, 1921, **21**, 1017). The Kremser theory is used to calculate the extraction of the hydrocarbon components in the absorber, while the Murray formula gives information on the volume and composition of vapours and liquid from compression of the vapours vented from the weathering of the fat oil and finally the quality and quantity of the gasoline and fixed gas.

The article deals chiefly with the Murray formula and gives the deviation factor to be applied to bring results obtained from its use in line with actual plant results.

A chart based on the Murray formula is given enabling calculations of the quantity and quality of liquid and vapour after compression and cooling to be made and an actual example is worked out. This is compared with the actual analyses of a plant run under the conditions given. C. L. G.

499 Notes on the Carbon-Black Flame. W. B. Wiegand. *Ind. Eng. Chem.*, 1931, **23**, 178–181.

Carbon-black or lamp-black differ in their mode of production in that the former is produced by impingement of the flame on a cold surface and the latter by collection of free soot. Carbon-black may be obtained from any self-luminous hydrocarbon flame (*e.g.*, from natural gas) which may be so "drafted" as to produce the best yield of the required grade of black.

A number of suggestions are advanced to explain the differences between carbon-black and lamp-black—*e.g.*, early fixation of the carbon particles in the flame leaves less opportunity for accretion; luminous carbon particles are positively charged and thus tend to keep apart, etc., these explanations possibly accounting for the finer particle size in carbon-black.

Two types of flame, round and flat, can be used, these differing essentially in what is termed their "combustion quotient," s/v, where s is the surface area of the flame, and v the volume of gases contained in the flame; s/v is greater for flat flames.

Several other factors besides "combustion quotient" affect the yield of black—*e.g.*, "drafting" (affecting both shape and size of flame), nature of orifice (affecting shape), and distance of impingement surface and gas pressure (both influencing size of flame). It is shown that optimum gas pressures and height of cooling surface exist for maximum yields. On the large scale, the size and shape of the burning house, the spacing, width and speed of the channels, and the spacing of orifices also affect results. The carbon-black obtained from round flames is generally greyer in colour, has a lower oil absorption, and mixes more easily in rubber, although it has less reinforcing effects in rubber compounds. E. B. E.

500. Many Economies Effected by Use of New Casinghead Gas Contract. E. C. le Fevre. *Oil & Gas. J.*, 1931, **29** (37), 189

For the past six months many companies have made use of the Standard Form of Casinghead Contract, developed by a Committee representative of oil-producing companies and natural gasoline manufacturers, appointed by the Natural Gasoline Association. The Contract Form provides an equitable agreement and is generally applicable.

The advantages claimed for it are: (1) it can be purchased ready printed, (2) it saves time in legal and executive departments, since modifications can only be made in spaces provided, (3) settlement is based on actual gross value

of gasoline content, and not on approximations to the nearest ½-gal. in gasoline content and to the nearest ½-c. in value per gal. of the gasoline, as in the department schedule or common modifications thereof.

In the departmental schedule, for example, the price of a gas with a nominal gasoline content of ½-gal. (*i.e.*, any gas under 0·75 gal.) is ¾ c. per 1000 cu. ft. for a range in price of 6 c. to 9 c. per gal. of gasoline. Assuming the nominal content, the price of the gas compared with the value of gasoline extracted varies from 16·66% to 25%. Working on these lines, a table of prices and percentages is given for gases with gasoline contents of ½ to 3½ gal., corresponding with values of gasoline from 6 to 15 c. per gal., as given in the departmental schedule. An average percentage is calculated for each gasoline content. The percentages used in the standard form agree with the average percentages of the departmental schedule for a gas of any given quality. This is recognised as a fundamentally sound principle. R. A. E.

501. Catalytically Oxidising Hydrogen Sulphide. A. G. Bloxam. E.P. 341,684. 22.1.31. Appl., 21.12.29.

This invention is based on the observation that complex compounds which are derived on the one hand from heavy metals, and on the other from cyclic organic compounds, have the property of accelerating to a marked extent the action of molecular oxygen on sulphuretted hydrogen and salts thereof. The invention is applicable for recovering sulphur previously absorbed from illuminating gas, coke-oven gas, and other industrial gases, containing hydrogen sulphide, the sulphur being recovered in elementary form or in the form of thiosulphate as a marketable product. H. E. T.

See also Abstract No. 505.

Motor Fuels.

502. Gasoline and Lub. Oils by Hydrogenation. R. T. Haslam and W. C. Bauer. *Oil and Gas. J.*, 1931, **29** (37), 34.

A brief outline of the history of the development of the hydrogenation process and the present-day methods of application is given. The oils produced after normal refinery treatment are practically speaking chemically pure hydrocarbons since sulphur, oxygen, nitrogen, asphaltic materials and other impurities are removed during the process in the form of gaseous products. The characteristic effects of the process are purification, stabilisation and homogenisation (*i.e.*, freedom from extremely complex structures of asphaltic or resinous nature, etc.).

A comparison is made between the properties of lubricating oils having viscosities at 210° F. of 75 and 62 secs. Saybolt prepared by hydrogenation (Hydrolubes), and those of high grade commercial oils prepared from Pennsylvania, mixed paraffin base, and Gulf naphthene base crudes. The results show that the Hydrolubes generally possess lower specific gravity, lower sulphur and organic acid contents and higher flash and fire points. The Conradson value is always low, especially when compared with paraffin base oils, and the rate of change of viscosity with temperature is relatively low and comparable with Pennsylvanian oils. Pour points in all cases depend on the nature of crude and extent of dewaxing.

The oils of 75 secs. viscosity were tested in a 4-cyl. White Motor truck engine, which was thoroughly overhauled before commencement, under carefully controlled conditions for 50-hour periods. Each hourly period

consisted of 50 min. on ¾ load at 1,400 r.p.m., and 10 min. idling at 450 r.p.m. beam load 25 lb. The room, outlet water and carburettor air temperatures were 70°, 185° and 100° F. respectively.

The Hydrolube was used in alternate tests so as to provide a basis of comparison to allow for changes in engine condition, at the end of each test the crankcase was drained hot, the remaining oil removed from engine and crankcase by washing with kerosine, the bearings and cylinder walls sprayed with the next oil to be tested. Carbon and valve gum were collected. The conditions represent those of moderate service.

Tests on the oils of 62 viscosity were made in a 6-cyl. Mack heavy-duty motor truck engine under more severe conditions, *e.g.*, room temperature of 100° F., fan and lower oil ring on piston removed, speed varied from 1,200 to 1,800 r.p.m. The test procedure was similar.

The results showed Hydrolubes to have the lowest consumption, and to give less carbon deposit and valve gum than any natural oil except in one case of a naphthene-base oil. The Hydrolubes showed to greater advantage in the more severe test.

Hydrogenated gasolines with octane numbers up to 95 have been prepared and a chart showing the relative effects of hydrogenated gasolines with octanes nos. 83·2, 87·7 and 90·4 and benzol on fuels of lower anti-knock value is given. Unlike cracked and str.-run gasolines, the heavier fractions of hydrogenated gasoline have a higher anti-knock value than the lower b.pt. fractions, thus indicating possibilities in the manufacture of high-flash aviation fuels of higher anti-knock value. Hydrogenated gasolines are more sensitive to addition of lead than some cracked and str.-run gasolines of similar octane number. They have S. contents generally below 0·03%, low gum contents and are stable to oxidation.

Much interesting information is presented on octane numbers, lead susceptibilities and the percentages of U.S.A. sales represented by gasolines of various octane numbers. R. A. E.

503. Vapour Phase Treatment. D. F. Gerstenberger. *Nat. Petr. News*, 1931, **6**, 59.

The relative merits of clay treatment of fractionated gasoline vapours from cracking still and acid treatment and redistillation of condensate are discussed. By preserving high content of unsaturateds it is claimed that clay treatment yields a gasoline with 14% higher anti-knock rating than acid refined product from same cracking stock.

Comparitive costs of the two processes are given and a saving of $0·2235 per barrel claimed for clay process. H. G.

504. On the Formation of Gum in Fuels. R. Brunschwig and L. Jacque. *Rev. Petrolifere*, 1931, (414), 311. (*Comp. rend.*, 1930, **191**, 1066.)

Experiments have been carried out to compare results obtained by the authors' method (*C.R.*, 1929, **189**, 486) with observations on various fuels stored more or less under normal conditions.

It was found that fuels stored in the dark, even with excess of air, did not show much increase in gum content; in sunlight, however, with a limited air supply, a considerable increase in gum content was noted, this effect being much greater when the air supply was increased.

This applied to motor benzols and commercial petrol both containing ethylenic hydrocarbons. Although the gum increased greatly in the benzols, in one case there was no corresponding darkening in colour.

A benzol with 147 mg. of gum in 100 c.c. was only very faintly yellow, while another with only 15 to 20 mg. was quite yellow. Fuels containing

unsaturated compounds, but giving satisfactory results both in storage and use, can form appreciable amounts of gum under the prolonged action of light and air.

Benzols of this type give satisfactory results in the authors' artificial ageing test which is comparable to commercial practice.

The development of colour or otherwise under the influence of light is no guide to the practical quality of a fuel. C. C.

505. Significance of New Specifications [Natural Gasoline]. S. S. Smith. *Oil and Gas J.*, 1931, **29** (37), 68.

Under the new specifications (see Abstract 183) the gravity of natural gasoline is omitted entirely and replaced by Reid vapour pressure and distillation limits. Under the old specifications the industry had in effect only one recognised grade for sale, *viz.*, A.A., for which the gravity limits were 80/87·9° Bé. and normally the gravity was as near as possible to 87·9° Be. and the distillation recovery close to the minimum of 90%. Grade 26-70 corresponds most nearly with the old grade A.A. and, on the assumption that gasolines will be made as close to the new limits as possible, the results of examination of a number of Mid-Continent natural gasolines, tested in the general research leading to the new specifications, are presented. The data is used to establish a relationship between vapour pressure and percentage evaporated at 140° F. on the one hand, and gravity on the other, which will serve as a guide to manufacturers who use natural gasoline for controlling the gravity of blends.

The A.P.I. gravity and percentage evaporated at 140° F. of the samples are plotted on a graph and show that for 70% evaporated the gravity lies between 85° and 89°, a range close to that of the old grade A.A. Another graph shows that only one of the samples having a vapour pressure of 26 or lower has a gravity in excess of 88° A.P.I.

Combining both series of results, it is estimated that the gravity limits of the new grade 26-70 will be within $\pm 2°$ of 86° A.P.I. for gasolines controlled closely to specification limits.

The new specifications will be useful to refiners who control volatility and vapour pressure according to seasonal demand, and it is hoped that they will foster an increased demand for natural gasoline for special purposes such as aviation fuels, high volatility motor fuels, etc. R. A. E.

506. Some Observations on the Composition and Recovery of Crude Benzol Produced under Different Carbonising Conditions. S. Tweedy. *Gas World*, 1931, **94**, 11-14.

Crude benzol may contain saturated aromatic hydrocarbons, paraffins, unsaturated hydrocarbons, sulphur compounds, nitrogenous bases together with varying amounts of wash oil and some naphthalene, the composition varying with carbonising conditions.

Colman's method of analysis is used. Typical benzols from horizontal retorts and also from coke ovens both contain about 61% benzene, 15-20% toluene and 10% xylenes, but the coke oven benzol also contains about 8% of heavy naphtha. Vertical retort benzol contains only some 32% benzene, 14% toluene, 24% xylene together with 15% paraffins. Comparative tests were carried out to determine variation in the composition of crude coke oven benzol produced at different carbonising times on two silica regenerative batteries of different type and capacity, also on two semi-silica batteries.

Durham coal was used containing 3% moisture (excepting one battery in which the Durham coal used contained 4·5% moisture) and 27–29% volatile hydrocarbons (dry basis).

The results of these experiments are shown in a table, which may be summarised as follows: (1) Coke oven benzol is a product of variable composition; (2) short carbonising times on the same battery increase the benzene content, with a corresponding decrease of the other constituents of benzol; (3) similar carbonising times on different batteries produce crude benzols of varying composition.

The paraffin content of benzol decreases with shorter carbonising times until it is present in very small quantities only. The effect of a suitable gas oil when used as an absorbent is negligible as regards paraffin content.

The chief factors which influence the composition of coke oven benzol are: (1) Type and size of oven, (2) carbonising temperature in the oven, (3) uniform heating of the oven, (4) full charge of coal in the oven, (5) class of coal, (6) moisture content of the coal, (7) operation of the oven.

It is found that the absorptive capacity of gas oil decreases rapidly with increase of temperature; a comparative table is given which illustrates this point, while curves are included showing the relation between relative rates of absorption and temperature. The efficient absorption of benzene above 20° C. is difficult. Scrubbing efficiency is of some importance in relation to the composition of crude benzol. W. A. W.

507. Methane as a Fuel. J. Bronn. *Brennstoff-Chem.*, 1930, **12** (2), 27-29, (3), 45-47.

Coke oven gas from the Ruhr province contains about 24% of methane and 4% of other gaseous hydrocarbons (ethylene, ethane and their homologues). This gas is decomposed by compression at low temperatures and the methane used directly as a gaseous fuel for automobile motors.

In the research described the methane contained about 80% of pure methane, 12-14% of ethane and ethylene and about 7% of CO, N_2, O_2 and H_2. The gas was kept in steel cylinders behind the driver's seat and a manometer registered the pressure. A homogeneous mixture of methane and air for combustion was added and regulated by means of a specially constructed mixing valve. Comparison tests in the same manner over the same distance and with the same speed showed consumptions of 1·26 cu.m. of CH_4, as against 1 litre of benzol for every 10 kms. driving distance. With methane the exhaust gas was found to be entirely free from carbon monoxide and contained very little unburnt methane.

The advantages and disadvantages of methane as a fuel are discussed. The fact that the gas has to be stored in heavy steel cylinders renders it suitable for use in heavy vehicles only. The application of the highly compressed gas in preference to liquid methane is recommended. It is quite unsuitable for export and is most useful for heavy vehicles in the Ruhr region where it is plentiful. W. S. E. C.

508. Non-knocking Motor Fuels. J. Y. Johnson. E.P. 341,068. 5.1.31. Appl., 3.10.29.

The production of an anti-knock blending material for gasoline is outlined. Aldehydes and alcohols containing more than three carbon atoms are catalytically reduced and the product, preferably topped to 180° C., is added to the fuel. Methanol synthesis by-products, "iso-butyl" oils, may also be so treated. H. C. R

See also Abstracts Nos. 562, 589, 591, 602.

Lubricants and Wax.

509. Effect of Carbon Black on Insulating Oils. W. B. Wiegand, C. R. Boggs and D. W. Kitchin. *Ind Eng. Chem.*, 1931, **23**, 273.

It is shown that oils, such as transil oil are improved as regards breakdown voltage, dielectric strength and power factor by treatment with thoroughly dried carbon black, such as, Micronex. Two methods of applying the carbon black were employed. In the first, carbon black was stirred into the oil and remained in contact with it for 24 hrs. with frequent agitation. The carbon black was filtered out through a layer of fine asbestos. In the second method, the oil was drawn through a 2-ft. column of dried screened carbon black in a glass tube. Ultramicroscopic examination showed that the filtered oil was optically empty. The asbestos filter is essential; filter papers introduce fibres into the oil. The carbon black appears to effect the thorough removal of moisture and a larger increase in breakdown voltage is produced by its use than by prolonged drying of the oil by metallic sodium. Agitation with carbon black followed by prolonged settling so that the oil only contained 0·0056 to 0·0073% of suspended fine black produced an increase of approximately 50% in the breakdown voltage.

The ability of carbon black (Micronex) to take up moisture from humid air, the effect of dispersed carbon black in oil on the power factor and the effect of carbon black additions on the power factor of suspensions of moist whiting in oil were also examined. In the latter case transfer of the water from the whiting to the carbon black occurs with resulting improvement in power factor. The electrical conductivity of carbon black is without influence.

R. W. L. C.

510. Potentiometric Determination of Acidity in Oils. R. N. Evans and J. E. Davenport. *Ind. Eng. Chem.* (Anal. Ed.), 1931, **3**, 82.

Comparative determinations of known acids dissolved in transformer oil have been carried out by the electrometric method and by alkali titration using phenolphthalein and alkali blue as indicators. Benzoic, acetic, stearic, cyclo-hexane carboxylic and a rosin naphthenic (abietic) acid were employed for the investigation. Low results were obtained in the estimation of stearic acid, in particular, using phenolphthalein as indicator and 50% methyl or ethyl alcohol as solvent. Concordant and satisfactory results were obtained for all these acids by the alkali blue method using a solvent composed of methyl alcohol and benzene and by the electrometric method.

The electrometric apparatus used has been described by Seltz and MacKinney (*Ind. Eng. Chem.*, 1928, **20**, 542 and *Ibid.*, Anal. ed., 1930, **2**, 1.

The silver-silver chloride electrode can be used as a reference electrode and this enables the agar-agar boundary, which it has been customary to use to separate the reference electrode from the solution under investigation, to be dispensed with. The electrometric titration curves show two points of inflection and the calculations are based on the first point of inflection. The main disadvantage of the electrometric method is the time required to carry out a determination. It is recommended that a preliminary approximate determination be carried out before the accurate estimation is made.

R. W. L. C.

511. FLUORESCENCE IN MINERAL LUBRICATING OILS. J. Tausz and A. Rabl. *Erdöl und Teer*, 1931, **7**, 106-107.

The fluorescence of mineral oils can be removed by the addition of a small amount of aromatic nitro-compounds. This is done technically in "deblooming." The authors have also found that the addition of very small amounts of gaseous sulphur dioxide or its solution will also produce this effect. By viewing a fluorescent oil through a very thick layer of sulphur dioxide solution, they conclude that the deblooming is not a physical action but depends on the addition of the sulphur dioxide or aromatic nitro-compounds to the fluorescent body by means of residual affinities. Picric acid also reacts with the fluorescent substances. The amount of SO_2 required is often only a fraction of a mg. for 1 c.c. of oil. They estimate the relative amounts of fluorescent substance present in a number of oils from various sources by titration of 1 c.c. of oil with a solution of SO_2 of known strength in toluol, until the fluorescence disappears. Typical results are: American refined lub. oil requires 6·3 mg. SO_2 for 1 c.c., Russian machine oil 1·57 mg., while various German oils need 0.63, 1·26 and 1·57 mg. respectively.

E. B. E.

512. CLASSIFYING TRANSMISSION AND REAR-AXLE LUBRICANTS. C. M. Larson. *J. Soc. Aut. Eng.*, 1931, **28**, 321-331.

Transmission and rear-axle lubricants are made from mineral oil with or without the addition of soda, calcium, lead or aluminium soaps; the soaps are added to decrease the tendency of the grease to leak from the housings.

It is considered that these lubricants should be classified in a similar way to mineral oils. A number of pieces of apparatus are described which are useful for making determinations of viscosity, viscidity, resistance to channeling, film-strength, etc. A number of greases have been examined by the methods described and results are given in graphical form and comparisons are made.

A pressure-viscometer was constructed for the determination of "pressure viscosities" at low temperatures. The grease is forced through a Saybolt orifice by means of air at constant pressure. The viscosity is given by Parsons and Taylor's formula: $PV = 31{,}500\ to/W$, where PV = pressure viscosity in Saybolt universal seconds, t = time in minutes, o = load in lb./sq. in. W = wt. grease forced out, in gms.

The viscidity of a grease is the quality of being viscid, adhesive and "ropy" and is more significant than the pour-point, which only indicates the consistency. An instrument has been designed for measuring this property and depends upon the resistance to a weighted spike of standard dimensions being forced into the grease at the required temperature. This gives a measure of the mobility of the lubricant, as well as its drag on gears. The amount of lubricant adhering to the spike and the distance it "strings out" on withdrawing the spike is a measure of the non-channeling and feeding qualities of the grease.

A "sleeve test" and a "transmission gearshift" test were also carried out to determine the resistance of the lubricant film and the relative shifting resistances in a Cadillac transmission system respectively.

By means of a machine embodying a pin driven at constant speed and subjected to increasing loads, the pressure a lubricant can be expected to stand without rupture can be measured, thus giving a means of determining the film-strength.

From the results given it is concluded that classification of lubricants in terms of viscosity and consistency only indicate their performance at low temperatures. Other qualities can only be estimated from practical tests.

The shifting effort in a transmission is proportional to the viscosity of the actual lubricant present independently of the body resulting from added soap content, except where channeling occurs.

The Saybolt viscosity at 210° F. can be used as an indication of the non-leak tendencies.

As regards film-strength, lubricants vary greatly, *e.g.*, one with standing pressures of 26,500 lbs./sq. in. without seizure while another failed at 7800 lbs. The addition of lead oleate or sulphur greatly increases the resistance to seizure, the latter being particularly effective.

As conditions of use of rear-axle and transmission lubricants vary so widely, no one detailed specification is applicable but consumers must be guided by a viscosity, consistency and film-strength classification which should be decided upon. E. B. E.

513. Formulas and Processes for Making Cold Set Rosin or Axle Greases. H. L. Kauffman. *Petr. Eng.*, 1931, **2** (5), 2.

Axle greases are made by mixing mineral oil, rosin oil and slaked lime in the cold, while cup greases are made from the lime soaps of fatty oil by boiling with mineral oil.

When considering the manufacture of cold set axle greases, care must be taken that the rosin oil is of satisfactory quality, so that the lime soap forms quickly and sets on mixing with the lime, and that the resulting grease is smooth and homogeneous. Rosin oils vary in viscosity, colour and free acid content, the latter property having great influence on the time taken for the grease to set. Laboratory tests are therefore carried out first to determine the setting quality, etc., of the rosin oil purchased. The mineral oil incorporated may be of practically any grade or quality from a pale 70-sec. oil to a heavy flux oil. Even slop oil may be employed in the cheaper types of greases. The lime used is usually of the standard quality for cup greases, for the sake of convenience in storing only one grade, but a cheaper grade could no doubt be used satisfactorily. Fillers such as graphite, ground cork, talc, etc., are often used, while cheap fatty oils are often incorporated in the rosin oil. The manufacture of cold set greases takes place in the following stages :—

(1) *Slaking the lime*, using approx. ¼-gal. of water to each lb. of lime in the kettle, for a period of from 5 to 10 min., after which the slaked lime is screened.

(2) *Preparation of the "set."* The "set" is an emulsion of pale oil and slaked lime, containing a small percentage of the calcium soap of rosin acids and a certain amount of free water. An average analysis is as follows :— Calcium hydroxide, 10·5%; free water, 8·5%; calcium resinate, 1·5%; mineral oil and unsaponifiable hydrocarbons from rosin oil, 79·5%. The set is manufactured by first removing the excess of water from the slaked lime by treatment with a small quantity of "main oil" (3 parts mineral oil to 1 part rosin oil). The rosin oil forms calcium resinate, which holds the lime in a fairly solid form, allowing the water to separate. The remainder of the mineral oil is then added.

(3) *Preparation of the main oil.* The main oil is prepared by blending mineral oil with rosin oil in the proportions necessary to produce the required degree of hardness, usually in ratio of 3–4 parts of mineral oil to one part of rosin oil. The rosin oil can be cheapened by adding "mixed oil" consisting of about 42% hard rosin, 18% first-run rosin oil, and 40% 30° Bé pale paraffin oil.

(4) *Mixing the set and main oil and drawing off.* The set and main oil are mixed in special machines as rapidly as possible, owing to the speed at which setting takes place. Details are given of the optimum operating conditions for each stage in the process. C. L. G.

514. Solubilities of Oils and Waxes in Organic Solvents. J. W. Poole and collaborators. *Ind. Eng. Chem.*, 1931, **23**, 170–177.

This work is a continuation of that previously published (*Ind. Eng. Chem.*, 1929, **21**, 1098), with the object of supplying information for the separation of paraffin wax from liquid hydrocarbons.

Solubility curves for the two component system wax-solvent are given for eight solvents. Of these ethylene dichloride seems to be perhaps most suitable, as it has a very low solvent power for waxes at low temperatures and is miscible with oil, in all proportions. *N*-amyl alcohol is suitable for dewaxing, except that it is too viscous to permit easy filtration at low temperatures. Methyl ethyl ketone and isopropyl alcohol have the disadvantage of not being sufficiently good solvents for oil.

The effect of oil on the solubility of wax in the three component system oil-wax-solvent is then considered, the results being given graphically. The effect of oil is generally to increase the wax solubility, in such solvents as methyl ethyl ketone, butanol, butyl acetate, etc.

Twenty-six further solvents were investigated qualitatively, and of these four, "pentasol" (commercial mixed amyl alcohols), paraldehyde, ethyl chlorocarbonate and butyl formate were examined quantitatively, as they appeared more suitable than most. Pentasol and paraldehydes seem to be among the best media for separation.

It is concluded that a very large number of solvents is available for dewaxing and suggested that the work might be extended, determining pour-points of the dewaxed oils instead of actual percentages of wax remaining in the oil. E. B. E.

515. Study of Wax from Low Temperature Tar. J. D. Davis and K. M. Irey. *Ind. Eng. Chem.*, 1931, **23**, 186–189.

The neutral oils from the tars produced by the low-temperature carbonisation of Mesa Verda (Utah) coal, contain appreciable quantities of a wax of high melting-point (54° C.). The wax was found to consist mainly of pentacosane (m.-pt. 54° C.) and heptacosane (m.-pt. 60° C.).

Various other bituminous and sub-bituminous coals were treated in a laboratory apparatus for low-temperature carbonisation. The tars from the former type did not yield more than 2% of wax, while those from the sub-bituminous coals gave 4·5 to 8·6%.

The method adopted for the recovery of the wax from the Utah tar was as follows. The neutral oil was separated from the tar by ether, tar acids removed by caustic soda, and bases of hydrochloric acid washing. The ether was then distilled off and the light oils removed by steam distillation. The residue was then refluxed with acetone and filtered at 5° C. After further recrystallisation the wax was still of a creamy colour, and turned dark brown on fusion. The melting-point was 55° C., the cooling curve indicating five different compounds. The other constants of the wax were $d\ \frac{20}{15}$ 0·9211; m.-pt., 55° C.; iodine value, 23; acid value, 1·1; ester value, 5·6. After purification by treating the ether solution with 95% sulphuric acid, an 85% yield was obtained of a white wax, m.-pt. 55·6° C., the cooling curve indicating three distinct compounds.

The wax is thus shown to be a pure dry wax of higher melting-point than marketable petroleum waxes. It is very suitable for the manufacture of candles, wax-paper, etc.

From the results with other coals it is seen that none of the true bituminous coals yield an appreciable amount of wax. The Utah coal is difficult to classify, but cannot be considered as a true bituminous coal. Carbonisation with superheated steam tends to increase the yield of wax. E. B. E.

516. Refinements of Mineral Oils and Mineral Fats. Compagnie Provençale de Raffinage de Corps Gras Mineraux. E.P. 342,834, 12.2.31. Appl., 12.12.29.

The process consists in dissol ving the mineral oil to be bleached in a solvent—namely, decahydride of naphthalene. 98% H_2SO_4 is then added little by little with strong agitation, maintaining the temperature between 25 and 30° C. The mixture is allowed to settle, the supernatent solution separated from the black acid, and finally treated in the usual manner with alkaline lye and fullers earth. The decahydride is separated from the solution obtained by a steam or vacuum distillation. W. S. E. C.

517. Distilling Hydrocarbon Oils. G. Cash, assr. to Standard Oil Co. U.S.P. 1,788,982, 13.1.31. Appl., 30.12.26. Also—P. L. Krauel, assr. to Standard Oil Co. U.S.P. 1,789,004, 13.1.31, 3.1.27.

These inventions relate to improvements in the distillation of lubricating fractions for the removal of their lighter constituents and the formation of a heavier lubricating fraction as a residium. The oil is passed in a confined stream through a heating zone, and then discharged into an enlarged chamber where separation of vaporised products takes place. The unvaporised oil is removed and forced into a stripping zone under an applied mechanical force, where it is treated with steam. Lighter constituents are removed thereby, and the mixture of steam and vapours forced into the stream of heated oil at an intermediate point in the heating zone. W. H. T.

518. Reclaiming Oils. L. D. Grisbaum. U.S.P. 1,791,474, 3.2.31. Appl., 12.12.25.

The dirty emulsion of oil and water is broken down by mixing it with a solution of caustic soda, although some other counter-colloid may be used, and thorough mixing is accomplished without mechanical agitation of any kind. By this means the emulsion is broken down, while greatly increased efficiency in the washing of the mixture and the separation of the impurities is effected. Subsequent dehydration of the oil is effected without the formation of an emulsion. Further objects of this invention include improved mixing and subsequent washing by passing the oil upwardly through the treating solution and then upwardly through a washing solution; accelerating and augmenting subsidence after treatment; spraying of the contents of the treating tank; the interposition of a layer of water between the cleaned oil and the subsided sludge; the utilisation of the force of gravity to aid in separating the impurities from the oil; and the purification of oils containing soaps without removing the soaps, as is now done in various mechanical processes. A further object of the process is to provide a system whereby large quantities of oil may be changed and cleaned at a time, and one which is substantially continuous for the charge, regardless of the quantity of dirt which it contains. H. E. T.

519. White Petrolatum. H. F. Gallagher, assr. to Standard Oil Development Co. U.S.P. 1,791,926, 10.2.31. Appl., 27.10.25.

The novel feature of this invention is embodied in the procedure adopted to produce a readily settling sludge on acid treatment. The process is carried out as follows: About 25 parts by vol. of crude petrolatum is mixed with about 37·5 parts of an asphalt containing petroleum residue, such as 40% bottoms from Cotton Valley crude, and about 37·5 parts of lubricating oil distillate. The mixture is treated with about 50% by vol. of fuming sulphuric acid, either in one batch or in successive portions, at 60–90° C. After the sludge has settled the treated oil is neutralised with dilute aqueous caustic soda and washed with a dilute aqueous alcohol, such as 50% -propyl alcohol. The alcohol is removed from the neutralised and washed product by steam distillation. An alternative procedure to produce a readily setting sludge consists in blending crude petrolatum with a heavy oil distillate, acid treating the blend as above described, and then adding about 30% by vol. of a preformed sludge, such as that obtained by treating white oil distillates with fuming sulphuric acid. R. W. L. C.

520. Treating Hydrocarbon Oil. R. E. Manley, assr. to Texas Co. U.S.P. 1,792,234, 10 2. 31. Appl., 15.4.27.

A process for removing paraffin wax from viscous hydrocarbon oils, which consists of mixing with the wax-bearing oil a suitable volatile carrier oil, derived, for example, from a naphthene base crude petroleum, less volatile than kerosine, and then distilling off the carrier oil together with the paraffin wax. The process is applicable to both batch and continuous operation. The process may also be operated so that the carrier oil, together with the volatile low-melting wax, is distilled off. The wax-bearing oil remaining is chilled to precipitate the less volatile high-melting wax, which is then separated from the oil. R. W. L. C.

See also Abstracts Nos. 491, 493, 502, 548.

Fluxes and Asphalts.

521. Bituminous Emulsions and Roads. B. Saladini. *Giorn. Chim. Ind. Appl.*, 1930, **12**, 583–587.

Three types of apparatus are used in the preparation of bituminous emulsions: (1) agitators or mixers, (2) an apparatus in which the liquids are forced between piston and cylinder walls of a suction pump, (3) colloid mills. Mechanical agitation alone is not sufficient: emulsifying agents must be added to stabilise the emulsion formed. The more usual materials belonging to this class are alkali soaps, resin soaps, etc., which give an emulsion of bitumen in water. Other reagents also used are gelatine, casein, naphthenic acids, etc.

Two typical formulæ are given, the finished product containing 48–50% bitumen, 49·5–45% water, and about 2% of emulsifier in each case. Details of the preparation are given, in one case the bitumen being heated to 95–98° C. and oleic acid added. Soda is added to water, which is raised to boiling and the two liquids then mixed. There appears to be an optimum velocity and time of agitation, while discontinuous agitation is sometimes more effective than continuous agitation. Details must be carefully carried out or coagulation

or inversion of the emulsion may occur—*e.g.*, excess alkali, dirty container, impure water, etc., all may have this effect.

Bituminous emulsions are dense liquids of a coffee colour, and should contain no separated bitumen or water. When spread in a thin layer on a surface, separation occurs and a film of bitumen is formed. Two photomicrographs are given of an emulsion before and after partial coagulation. Certain tests on these emulsions are described, and the usual tests are carried out on the bitumen recovered from the emulsion.

The more interesting tests on the emulsion are those which give some indication of stability—*e.g.*, a tube 20 mm. in diameter is filled with emulsion, and the stability of the latter is measured by the height of the clear layer on standing for periods of from 3 days to 3 months. For a second test a slab of basalt is suspended in the emulsion, and after draining in air the surface is sprayed with distilled water and the time of breaking of the emulsion as indicated by colouration of the water noted.

Graefe's Ring test is also described. The emulsion is placed in a ring on filter paper, and the time taken for the water to reach an outer concentric ring is noted.

An emulsion should be sufficiently stable not to break down during storage or transport, but must not be too stable to prevent breaking occurring when the emulsion is applied.

On application to a road, the water partly evaporates and partly diffuses through the surface of the road, leaving a thin layer of bitumen. These emulsions are much less costly to apply than hot asphalt or bitumen, and they are practically independent of weather conditions.

Other uses of bituminous emulsions are noted, most of which are based on the formation of a protective coating on drying.

A short note on the advantages and disadvantages of these emulsions is included, the latter being particularly suitable for roads with light traffic, as it gives an impermeable, dust free and elastic surface without the use of costly or complicated machinery. C. C.

522. Experimental Mixed-in-Place Macadam Road. *Roads & Road Constr.*, 1931, **9**, 99–100.

This is a description of the experiments carried out by the N.S.W. Main Roads Board (Australia) with a mixed-in-situ macadam pavement which have met with considerable success. Seven sections of road, 24 ft. wide, were dealt with in different ways. Common to all was the following procedure: loose dust was removed by horse brooms and a course of 1½-in. metal, one stone in thickness, was distributed from end-tipping lorries. A little raking was followed by the superimposition of ½-in. metal at the rate of 1 ton per 19 sq. yds. of road surface. Tar was then applied by mechanical sprayers operating at a nozzle pressure of 40 lbs/sq. in. Mixing of the metal and tar was effected by an eight-foot grader of the leaning wheel type. After mixing, the metal was graded and shaped to the desired section, and each section was then subjected to rolling and compacting by different methods. The most important conclusions drawn from these experiments are: application of a primary coat of tar to the gravel base is essential. Without it, there is a lack of adhesion between the pavement and the gravel. Following final shaping with the grader, better results are obtained by using a light roller only. After a preliminary rolling, consolidation can be effected safely by the traffic. At the proper time final rolling with a 10 to 12-ton roller should be carried out. This mixed-in-situ process can provide at reasonable cost a satisfactory pavement which should serve as a useful intermediary between tar or bitumen surfaces roads and penetration macadam pavements. C. I. K.

523. Significance of Surface-Testing and Control of Materials in Asphalt Road Making. R. Wilhelmi, *Erdöl und Teer*, 1931, **7**, 78–79, 92–93.

A knowledge of the nature and properties of the materials used is necessary to avoid faults in the construction of modern roads. The quality of the materials is governed by specifications (DIN. 1995 and 1996, and DIN. DVM. 2101–2103), which form the basis for evaluation of such materials in Germany.

In America extensive tests are carried out on the actual work to control sizes of sand, etc., penetration of the asphalt employed, etc.

The viscosity of the tar is an important means of judging its suitability, and can be easily and quickly determined on the work by means of Mallison's Tar Consistometer.

Test-pieces of the road should be removed after laying and subjected to mechanical and other tests, from which the composition can be controlled and the resistance to wear and deformation by traffic can be estimated. The details of carrying out some of the tests are discussed. E. B. E.

524. Road Surfaces and Mechanical Recording of their Condition. P. E. Spielmann. *Roads & Road Constr.*, 1931, **9**, 76–78.

Mechanical recording of road surfaces aims at the obtaining of a numerical measure of the state of the road surface so that comparisons may be made at a later date in order to estimate the nature and extent of wear and tear. Brief descriptions and references are given of the Italian "Profilometer" (a metal frame on 16 wheels with a central indicating mechanism actuated by a small wheel in contact with the road surface) and the Danish "Viagraph" (a sledge-like machine). These and similar types fail in as much as they measure the condition of a road with the surface itself as a datum level. A second type of Danish instrument ("Bridge") is of the nature of a straight edge which bestrides the road and rests on special fixed points, so that a general wear over the whole surface can be measured. Difficulties which might arise with such measurements from the effects of movements due to subsidence and temperature changes in concrete are being overcome in Italy by embedding steel cylinders to a level below the road surface and protecting them by a plug of asphalt, until they are required as indicators of datum level.

The American Roughness Indicator is fitted to the chassis and front wheel of a motor car, and measures the amount of bumping in inches by means of a counter. This type offers a quick method of estimating the value of a contractor's work as well as the different action of the traffic on the road.

The German Static Accelerometer is a sort of highly specialised seismograph which records bumps as an acceleration in meters/sec^2. C. I. K.

525. Highways of the U.S.A. Current Constructions, Types, Methods and Costs. Part IV.—*Bituminous Penetration Method.* T. W. Allen. *Roads & Road Constr.*, 1931, **9**, 64–65.

Waterbound macadam widely used before the development of heavy automobile traffic is not well suited to present-day conditions because rubber tyre suction removes the binder and causes ravelling. Bituminous surface treatment was therefore used, but this has not been satisfactory, because traffic not being uniformly distributed causes uneven wear. Intensely used areas, therefore, required frequent treatments, which cause a building up of the mat on the less worn areas, and this leads to unevenness of surface due to the mat creeping.

Penetration macadam retains the advantages of a macadam pavement and avoids the disadvantages of the two types mentioned above. It is used extensively in the States, because it is a surface which yields complete highway

service. Usually either gravel, or telford, or rock sub-base is used to strengthen the base course, which has to be adequate, substantial and free from settlement; this accounts for the rather heavy expense of the method. The crown of the base course must be correct and uniform, and much attention should be paid to the provision of an adequate surface on the base course before the penetration top is laid down. A base satisfying the above requirements is covered with hard, tough, dust-free stone, all of which should be retained on a 1½-in. screen (but smaller than 2½ in. to 3 in.) and a considerable portion should be retained on a 2½-in. screen if the finished thickness of this layer is to be between 2½ in. to 3 in. It is thoroughly rolled by a 10-ton roller in order to key the stones together and to key the larger pieces into the base. The surface should be checked for smoothness during rolling. Correct distribution and controlled rolling give a smooth surface, which is then treated from a pressure distributor with the bituminous material of about 100 penetration, at a temperature of from 325° F. to 375° F., and at the rate of 1½ gal. per sq. yard. Then ¾-in. to 1½-in. stone is uniformly distributed to fill the voids, which is then rolled and finally swept clean. A second application of bitumen at the rate of ½ gal. per sq. yard is followed at once with dustless stone chips ¼ in. to ¾ in. in size, which are thoroughly rolled in. A surface of this type, exclusive of the cost of the base, costs from $5000 to possibly $7500 per mile of 18-ft. roadway. C. I. K.

526. Highways of the U.S.A. Part V.—*Bituminous Asphalt Pavements.* T. W. Allen. *Roads & Road Constr.*, 1931, **9**, 87–89.

The 13,000 miles of bituminous pavements on the rural roads in the U.S. are being added to at the rate of about 850 miles per annum at an average cost of $30,000 per miles for constructing the surfacing and foundation. Native and petroleum asphalts and refined coal tars are mainly used in such bituminous pavement construction; only very small amounts of either sandstone or limestone rock asphalt are used in the U.S. for this work.

The sheet asphalt, modified Topeka and bituminous concrete are representative class types of most of the bituminous pavements in the U.S.A. These classes may be differentiated on the basis of the maximum size of the permissible aggregate. Standard sheet asphalts use only mineral aggregate which passes a No. 10 sieve (0·079-in. opening). Modified Topeka mixes use aggregates grading from dust to that which passes a ½-in. screen, and bituminous concrete types permit the use of aggregates grading from dust to that passing 1½-in. to 1¾-in. screen. These types have common characteristics. The aggregates are so chosen as to give a mass possessing a high degree of natural stability and low void content. Aggregates except dust are heated to a high temperature, mixed in a pug mill with a known amount of bitumen suitable for the traffic and climatic requirements. Mixing takes place over a period of from 40 to 60 seconds, and the hot mixture is transported to the road in trucks, spread and compacted while still retaining sufficient heat to maintain a certain degree of plasticity.

These types also require a substantial foundation. Old existing roads are frequently used especially for bituminous concrete. The compacted thickness of the wearing course alone varies from between 1½ to 2 ins., and the width of the surfacing where these types of pavement are used is rarely less than 18 ft.

Several tables are given of the typical gradings for the representative bituminous pavement types, together with a lengthy discussion of the economics and business aspects of the subject. The latter is warranted by the fact that the way in which the work is or can be performed has a rather surprising influence on the cost of the completed pavement. C. I. K.

527. Asphalt Shingles. J. M. Weiss. *Ind. Eng. Chem.*, 1931, **23**, 168–170.

The use of asphalt shingles for roofing is important. Ninety per cent. of those sold are of the strip type, about 32 ins. or 36 ins. long, 12½ ins. wide, with cut-outs 4 ins. deep and ½-in. wide. With a 4-in. exposure to the weather, 311 sq. ft. of actual shingle will cover 100 sq. ft. of roof.

The shingles are made from roofing felt (rag paper with or without other fibres) saturated with asphalt and covered on the weather side with an asphalt coating in which mineral granules (usually slate) are embedded.

Tests were made by the author on 16 marketed brands of shingle. Only one of these failed to comply with the requirements of A.S.T.M. Specification D225–29. The author considers more rigid purchase specifications are desirable. An upper limit should be fixed for the wood-fibre content of the felt (one sample was found to contain 46%) as, although wood fibre is much cheaper than rags, the results are inferior.

The recommended saturation of felt with asphalt is 170% minimum, against the A.S.T.M. minimum of 150%. It is also suggested that a ratio should be prescribed between the amount of surface coating and mineral matter. Mineral-coated shingles are generally more resistant to wear, but enough asphalt coating should be used to prevent the grit becoming easily detached. Exact recommendations are not made on these points. E. B. E.

528. Combating Imported Bitumen in Australia. Progress in the Production of Bitural. R. J. Bennie and H. E. Groves. *Gas World*, 1931, **94**, 185–187. (From a paper presented at the Victorian Institute of Engineers, Melbourne.)

Bitural is a new road binder, the product of research carried out at Melbourne. By polymerisation and molecular condensation the character of the unsaturated hydrocarbons and tar acids in gas works tar is changed, producing gummy resinous bodies with the properties of good road binders.

The polymerisation is achieved by passing hot air through a 12-ton charge of the tar contained in reactors at a rate of 500–600 cu. ft/min., the treatment being completed in digesters, where dehydration and removal of recovered oil takes place, the process taking 48 hours.

From 2500 gal. of tar is produced 1800 gal. of bitural of 160° of penetration; also about 700 gal. of recovered oil boiling between 170–300° C.

The bitural is suitable for road and path material, bituminous paints, etc., and the oil is useful as fuel or working up into creosote-oil, phenyle, fruit-spraying oil, etc. Details of plant dimensions, temperatures and other economic factors are given. W. A. W.

529. Manufacturing Dispersions. H. D. Elkington. E.P. 341,443, 9.1.31. Appl., 9.10.29.

Dispersions of bituminous materials such as asphalt, tar, pitch, etc., are prepared with the aid of dispersing agents. These consist of finely divided solids which are insoluble in the dispersing medium, and contain added electrolytes which are rust-inhibitive substances such as chromates and bichromates. The dispersions are then subjected to a heating action during the gradual addition of one or more electrolytes in amounts as would cause the dispersion to flocculate if added to the dispersion before the heating action. The heating action is continued after the addition of the electrolyte is completed. W. S. E. C.

530. Manufacture of Bituminous Binders for Road, Stone and the Like. South Metropolitan Gas Co. and H. Pickard. E.P. 341,901, 23.1.31. Appl., 23.7.29.

Coal is dispersed in tar at a temperature of 250–300° C. to the extent of 10–18% of the weight of the tar. This tar-coal preparation is mixed with a further quantity of pitch not exceeding two-thirds of the weight, and the mixture blended with a sufficient quantity of suitable mineral matter to constitute between 20 and 60% of the final mixture. W. S. E. C.

531. Treating Aqueous Dispersions of Bituminous Substances. *N.V. de Bataafsche Petr. Mij.* E.P. 341,914, 21. 1. 31. Appl., 21.10.29.

Aqueous soap dispersions of bituminous substances are mixed with asbestos, lampblack, acid pigments and other similar substances which normally tend to break the dispersions. These substances are first treated with an alkali hydroxide or trialkali phosphate in quantities sufficient to alter their hydrogen ion concentration to a value at which their capacity to break the dispersion is substantially neutralised; the treated substance or substances are then admixed with the dispersion. W. S. E. C.

532. Manufacture of Dispersions of Bituminous Substances. *N.V. de Bataafsche Petr. Mij.* E.P. 342,031. 29.1.31. Appl., 15.11.29.

This invention relates to a method of producing aqueous dispersions of bituminous substances which are normally immiscible with water. The dispersing means consists of mixtures of mineral paste-forming colloids in quantities which are relatively small compared with the quantity of bituminous material treated, and of surface-tension lowering agents in aqueous suspension, and in amounts which are relatively small compared with the quantity of paste-forming colloids used. The bituminous materials are then added in the molten condition. W. S. E. C.

533. Aqueous Dispersions of Bitumen. H. D. Elkington, assr. to *N.V. de Bataafsche Petr. Mij.* E.P. 342,296, 26.1.31. Appl., 25.10.29.

A description is given of a process for manufacturing aqueous dispersions of bituminous materials with the aid of solutions of gelatine in water. Gellatine inhibitors in the form of water-soluble metallic salts are added in amounts varying between 25 and 100% by weight of the gelatine to the solutions prior to or during admixture with the bituminous materials. W. H. T.

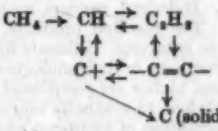
Special Products.

534. Mechanism of the Effect of Electrical Discharges on Methane. K. Peters and O. H. Wagner. *Brennstoff—Chem.*, 1931, **12** (4), 67–68.

The reactions concerned are: (1) $2CH_4 = C_2H_2 + 3H_2 - 92$ Kal.; (2) $2CH_4 = C_2H_4 + 2H_2 - 55{\cdot}6$ Kal.

Measurements were taken of the maximum temperature in the discharge vessel and it was observed that the colour of the discharge changed spontaneously from light blue to yellow in the region of mean discharge, and this indicated an alteration in the mechanism of the reaction. The temperature increased with the charge.

Spectrum analyses of the electric glow before and after the colour change were next investigated. The reaction is assumed to take place according to the following :—



Before the colour change occurs CH lines are present and small quantities of *N* and CN lines. The CH_4 molecule appears to be decomposed first to CH_3, then CH_2 and finally to CH radicles, which form polymers of saturated hydrocarbons at low pressures and with small charges; C_2H_4 and C_2H_2 form at high pressures. In the region in which the colour change takes place 2CH groups are observed with high charges; then a —C=C— radicle forms, which reacts with hydrogen. High charges favour the formation of —C=C— radicle and decomposed acetylene produces solid carbon. After the colour change —C=C— lines, CN, CH and C+ lines are observed. W. S. E. C.

535. Specific Heat of Diphenyl. H. O. Forrest, E. W. Brugmann and L. W. T. Cummings. *Ind. Eng. Chem.*, 1931, **23**, 37–39.

These workers used two different methods for determining the specific heat and plotted a curve showing the relationship between specific heat and temperatures ranging from 77·6° C. to 347·0° C. C. I. K.

536. Specific Heat of Liquid Diphenyl. R. F. Newton, B. D. Kaura and T. De Vries. *Ind. Eng. Chem.*, 1931, **23**, 35–37.

The commercial production of diphenyl has made possible its use as a medium for heat transfer. This paper describes various methods used to measure the specific heat of the liquid. From the results obtained the following formula was derived and is believed to hold from its melting point to 300° C. to within ±1%:—

Specific heat of liquid diphenyl = 0·388 + 0·00057 t, where t = temperature in °C. C. I. K.

537. Production of Carbon by the Dissociation of Gases. S. Percival, Assr. to Chr. Hostmann-Steinberg'sche. E.P. 340,235, 12.12.30. Appl., 12.9.29.

The production of carbon is accomplished by splitting up gases which have a tendency to dissociate at high pressures, such as acetylene. Pressure is supplied by a special compressor from which the gases are charged into the reaction tubes and ignited. The excess pressure produced is reduced by blowing off hydrogen into a pressure vessel. Later in the process this hydrogen is used to tear the carbon from the walls of the tubes. By means of an exhauster system the tubes are blown through, or scavenged, by a mixture of hydrogen and soot. E. N. H.

538. Polymerisation of Olefines. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 340,513, 22.12.30. Appl., 20.9.29.

A statement is given of certain catalysts suitable for the production of polymers from the lower olefine hydrocarbons at temperatures between 200° C. and 400° C. For this purpose phosphoric acid, metaphosphoric acid, and other acids of phosphorus, or salts of these acids are suggested, provided

the catalyst is in a solid, or "solid-like" state. Alternatively, it is claimed that compounds which furnish such acids of phosphorus, under the conditions of the reaction, such as esters of these acids, or the oxides which form the basis of the acids, may be used. If desired, carriers, such as Florida earth, charcoal, silica gel are suggested. The reaction products, it is stated, do not form double compounds with the catalysts. Diluents like water vapour, hydrogen, nitrogen, carbon dioxide, methane or its homologues may be present. Pressures up to 1000 atmos. and even higher are mentioned. By adjusting conditions, products varying from butylene to synthetic lubricating oils may be obtained without the substantial formation of hydrogen and methane, or deposition of carbon. When the polymerisation is carried out in a closed vessel mixed products are obtained, containing gasoline-like products, middle oils and lubricating oils. The gasoline-like products, it is claimed, are proof against knocking to a high degree when employed in internal combustion engines. Yields up to 93% are claimed. E. N. H.

539. Purification of the Gases Containing Acetylene Obtained by the Treatment of Carbonaceous Substances in the Electric Arc. I. G. Farbenind. A.-G. E.P. 340,787, 8.1.31. Appl., 6.1.30.

Gases containing acetylene obtained by treatment in the electric arc may be purified by passage over active porous masses. This it is stated removes di-acetylene and other strongly-colouring substances, and if necessary the gases may be freed from cyanogen compounds before or after this treatment. After purification the gases are suitable for working up into conversion products of acetylene. E. N. H.

540. Polymerising Unsaturated Hydrocarbon Gases. C. R. Wagner. E.P. 341,391, 8.1.31. Appl., 8.7.29.

Polymerisation of the unsaturated gases containing ethylene and its homologues is brought about at temperatures between 800° and 1000° F. by application of pressure between 300 and 1000 lb./sq. in. The chief object is to utilise the gases produced during pyrolytic cracking operations for the production of hydrocarbons suitable for use as motor fuels. The process may be carried out with, or without, catalysts such as iron oxide. Since the reactions are of an exothermic nature, the furnace is so arranged that the heating gases flow parallel with, and in the same direction as, the gas to be treated. By this means a balance is obtained and it is claimed temperature control is good. The products obtained are cooled and condensed without releasing the pressure. The lean gases are separated and may be used for firing the furnace. The condensed liquids are stabilised and the volatile hydrocarbons so removed are returned to the reaction zone. At temperatures above 1000° F. the formation of carbon becomes troublesome and the yields of liquid hydrocarbons decrease. E. N. H.

541. Decomposition under the Action of Heat of a Mixture of Methane or Other Hydrocarbons and Water Vapour. Union Chemique Belge, S.A. E.P. 341,393, 5.1.31. Appl., 3.9.29.

By the application of heat in the absence of catalysts hydrogen and carbon monoxide may be produced from a mixture of water vapour and methane, or gases containing gaseous or volatile hydrocarbons. The endothermic decomposition is carried out using two separate circuits in a stationary heat exchanger. In one circuit is the gas to be treated and in the other the gases supplying the heat. The heat exchangers may be operated in combination with heat regenerators. E. N. H.

542. Producing Condensation Products from Polybasic Acids and Polyhydric Alcohols. H. A. Bruson and K. Albert G.m.b.H. E.P. 341,447, 12.1.31. Appl., 11.10.29.

Aliphatic polybasic acids possessing more than six carbon atoms linked together in a normal straight chain, in particular sebacic acid, are heated separately or in mixtures with di-, tri-, or polyethylene glycol separately or in mixtures. The condensation products formed are mixed with nitrocellulose or other cellulose derivatives and organic compounds are obtained which are tough, flexible and elastic. Natural or artificial resins, especially phenol-aldehyde resins, may be added to the mixture when mixed esters are formed.

W. S. E. C.

543. Manufacture of Rubber-like Products by Modifying the Physical Properties of Organic Isocolloid Materials Containing Unsaturated Carbon Compounds. I. L. Auer. E.P. 341,490, 8.1.31.

A modification of the process claimed in E.P. 318,562 for the manufacture of rubber-like products from fatty products is described. Certain products manufactured according to specification No. 318,562, for instance, fatty oil emulsions treated with a gas as modifying agent, have rubber-like properties, *i.e.*, they may be plastified by treatment on the rubber mill and can be mixed with various compounding ingredients used in the rubber trade. After treatment with the modifying agent—an agent which is neither a gas nor a gas-liberating substance may be used in addition—one or more non-rubber constituents of natural rubber latex are added to the modified substance or to the emulsion obtained. The precipitable substances are then precipitated or flocculated on the globules of the emulsified fatty substance by addition of precipitants for these substances. Stearic acid is added wholly or in part to the fatty substances used as starting materials before preparation of the emulsions, as the stearic acid is fusible with or soluble in the fatty substance without decomposition. The product obtained is subjected to an after-treatment at a suitable temperature with sulphur, sulphur chloride or other vulcanising agents. The aqueous medium is separated from the transformed fatty substance by coagulation, evaporation, electrophoresis, ultra-filtration or the like. Alternatively, admixture with other emulsions may be first effected and the aqueous medium is then removed whereby a homogeneous mixture of the transformed fatty substance and other emulsion is obtained. Exposure to radiant energy (X-rays, ultra-violet rays, etc.) may assist any of the described processes.

W. D. S.

544. Heat Treatment of Hydrocarbon Gases. Soc. d'Etudes et Realisation dite "Ereal." E.P. 342,034, 29.1.31. Appl., 18.11.29.

Carbon in the form of lamp black may be produced by passing gases containing hydrocarbons such as methane through an incandescent fuel material like coke at temperatures between 800° C. and 1200° C. By the application of injection or suction, or both, the carbon is carried along with the cracked gas and is separated either by expanding the gases or by an electrical method. During the blowing period, air, mixtures containing oxygen, pure oxygen, or mixtures of a gaseous oxidising agent and steam may be used. A system of heat recuperators is used.

E. N. H.

545. Substitution Products of Aromatic Hydrocarbons. W. W. Groves and I. G. Farbenind. A.-G. E.P. 342,373, 5.2.31.

Substitution products of aromatic hydrocarbons are obtained by treating the latter with cyano-acetyl chloride by the Friedel-Craft's reaction. The condensation can be applied to all aromatic hydrocarbons such, for instance,

as those of benzene, naphthalene, anthracene, acenaphthene series. Preferably the reaction is carried out at a temperature not exceeding 60° C., for instance, 30–45° C., in the presence of an acid-condensing agent and a diluent or a solvent. The products are valuable intermediate products for the manufacture of dyestuffs. Several examples are given. W. D. S.

546. Solutions of Salts of Sulphuric Acids. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 342,399, 5.2.31. Appl., 11.11.29.

A process is described for increasing the dissolution in water of water-soluble salts of sulphuric acids (referred to as fire-extinguishing agents in specification No. 319,083). The water or the salts is incorporated before or during dissolution with urea or thiourea. W. H. T.

547. Production of Diolefines. J. Y. Johnson and I. G. Farbenind A.-G. E.P. 342,411, 5.2.31.

Butadiene or the other corresponding diolefines are obtained from 1-3-butylene glycol or other 1-3-dihydroxy paraffins such as 2-methyl-1-3-butylene glycol by leading these compounds over catalysts. The catalysts are of the type which favour the splitting off of water at elevated pressures of from about 2 to 20 atmos., preferably at about 10 atmos., and at temperatures of from about 270° to 300° C. Examples of such catalysts are phosphoric acid, red phosphorus, sodium phosphate, potash alum, aluminium oxide activated with a little copper phosphate, aluminium phosphate, thorium oxide. If desired these catalysts may be precipitated on to carriers. The advantage of employing the elevated pressure lies in the fact that a greatly increased throughput is obtained without any substantial decrease in percentage yield. Too high pressures may, however, cause a decrease in yield. Examples are given. W. D. S.

548. Refining of Fatty Acids Obtained by the Oxidation of Paraffin Wax and Other High Molecular Organic Compounds. J. Y. Johnson. E.P. 342,670, 23.1.31. Appl., 23.10.30.

The products obtained in the oxidation of high molecular organic compounds with higher nitrogen oxides or nitric acid, are heated and simultaneously distilled to a temperature below 300° C. in the presence of hydrogen and of a hydrogenation catalyst above atmospheric pressure if desired. W. S. E. C.

See also Abstracts Nos. 507, 595.

Refining and Refinery Plant.

549. Acid Treatment Process. H. A. Young, *Mon. Petr. Roumain*, 1931, **31**, 235–237.

Acid treatment of oils should be carried out at as low a temperature as possible; raising the temperature increases the sulphonic acid content of the oil. Mechanical agitation is preferable.

A technique has been developed which consists essentially in the addition of certain porous solid materials which have a known force of attraction for the liquid or gaseous molecules.

A preliminary short period of standing facilitates the removal of the heavy impurities.

"Hyflo-Supercel," a white inert, siliceous powder has been found to be satisfactory.

A mixture of Hyflo and distilled oil is pumped on to the filter. The acid oil, to which is added more Hyflo, is pumped through this. A minimum time of contact is required but optimum conditions can be determined in the laboratory. The filtration is carried out at the same temperature as the acid treatment.

Usually, oils are treated with acid in three stages—a period of settlement being allowed after each step, earth treatment following the last acid wash.

For oils of viscosity 5·5° E. about 0·3—0·5% Hyflo is required while bright stocks need 0·7—1·0%.

About 4% of the heated oil is retained by the earth. It is claimed that up to 92% of the acid is removed by filtration and that the resistance to emulsification is much increased. In addition, less of the neutralising material is required after the Hyflo, while there is a reduction of 20% to 40% in the total amount of earth required, and the process is more economical, generally, than other methods. C. C.

550. Action of SO_2 on Mineral Oil. A. L. Forster. *Nat. Petr. News*, 1931, **23** (7), 63—73; (8) 60—74; (9) 73—78.

A résumé of the literature and an indication of the steps in the commercial development of the Edeleanu process is given. Modern plant for continuous treatment of paraffin base and naphthene base oils is described. Paper covers theory and range of operation of the process, details of operations and costs. Properties of materials before and after treatment discussed to some length. Claims for process are: (1) Simplicity of operation. (2) Absence of chemical action. (3) Material extracted may be used for profitable purposes. (4) Solvent is cheap, non-corrosive under operation conditions and 100% recoverable. (5) Process is continuous. (6) Filtration through clay, only subsequent treatment necessary. H. G.

551. Regenerating Litharge for Sodium Plumbite Doctor (Treatment). *Chem. Met. Eng.*, 1931, **38** (2), 76.

A recent interesting process has been developed for the recovery of spent doctor solution and its conversion from alkaline lead sulphide back to sodium plumbite solution. The chemical reactions involved are: (1) $2\,PbS+2O_2+2NaOH=2\,PbO+Na_2S_2O_3+H_2O$; (2) $2\,PbO+4\,NaOH=2\,Na_2\,PbO_2+2H_2O$.

The plant described recovers daily approximately 17,000 lb. litharge, 55,000 lb. caustic solution and 450 barrels oil, using two million cu. ft. of air, and is operated by one man per shift of eight hours. The spent material containing 0·6% PbO, 11% caustic in solution and 4% PbS as sludge, is allowed to settle in two 85,000 barrel storage tanks, used alternately and 300/400 barrels of oil recovered. The middle sludge fraction containing solid PbS in an emulsion of caustic and oil, is transferred to a "break tank" and heated to 150° F. by steam coils. Separation occurs and may be assisted by a small proportion of sulphur flour, yielding layers of oil, caustic solution and solid lead compounds. The oil is removed (90/160 barrels per day) and the remainder transferred to two blow tanks of 32,000 gal. capacity, where it is mixed with sufficient caustic solution from the storage tank to make up to 24,000 gal., heated to 175° F. and blown with air for eight hours. About one-third is then removed and replaced by solution or suspension, as required. The recovered solution is mixed in the proportion of two to one with the oil from the blow tanks in an orifice plate mixer, to remove the coloured matter present, and is then ready for use again. When the concentration of sodium

sulphite reaches 20/25% retardation of oxidation and incrustation of pipes occurs, so a portion of the caustic solution remaining in the storage tank is removed from the system and used as a neutralisation wash. The make-up only amounts to 6/7% calculated on a litharge basis. A flow sheet of the process is given. R. A. E.

552. Apparatus for Carrying Out Distillation Processes. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 342,741, 9.2.31. Appl., 9.8.28.

A process is described for the distillation of high-boiling liquids under reduced pressure. During distillation a gas, under reduced pressure, is circulated through the body of the still connected with the gas space of the receiver and thence through a pipe leading into the liquid within the still. A gas circulation pump is provided together with means for establishing reduced pressure in the apparatus. W. H. T.

553. Heat Exchange Apparatus. A. D. Harrison. E.P. 342,959, 12.2.31. Appl., 10.4.30. Conv. (U.S.A.), 10.4.29.

Claims are made for a heat exchange plate apparatus in which heat is communicated from fluid to fluid through plates provided on both sides with endless flanges. The latter form uninterrupted packing rims entirely surrounding bridge ports and sinuous grooves in the plates forming passages for the liquids. The plates are assembled in numbers with a separating plain plate between each pair to close the sinuous passages on the grooved plates. W. H. T.

554. Purification of Oil. L. H. Clark. U.S.P. 1,788,911, 13.1.31. Appl., 16.12.25.

This relates particularly to a method and apparatus for the renovation of oil that contains some or all of the following: products of decomposition, free fatty acids, water, soaps, sludges which usually have an acid reaction and which are insoluble in the oil, finely-divided carbon and other impurities. The process consists in first mixing the oil with an aqueous solution of an alkaline reagent such as sodium silicate, sodium aluminate, tri-sodium phosphate or caustic soda and soda ash, separating the impurities and the aqueous phase of the resulting mixture from the oil, then heating the oil and passing another liquid into heat exchange relation with the heated oil, and thereby producing vapour and bringing it into contact with the heated oil for the furtherance of the evaporation therefrom of contaminating liquids in solution. After steam refining the oil it is passed through a centrifugal machine to remove immiscible impurities. H. E. T.

555. Refining Oil. A. P. Bjerregaard. U.S.P. 1,791,521, 10.2.31. Appl., 26.8.25.

A process of treating gasoline is described in which traces of water are first removed and then an addition of less than 1% of methyl alcohol is made. The mixture is then contacted with dry alkali hydroxide and finally separated. W. H. T.

556. Use of Entrainment Separators in Vacuum Distillation. A. C. Spencer, Assr. to Standard Oil Development Co. U.S.P. 1,791,940, 10.2.31. Appl., 1.10.26.

The vapour stream from the vacuum distillation is cooled without substantial condensation and then the direction of flow of the stream is changed suddenly. The linear velocity of the vapour stream is thus decreased and the vapours passed to a condensing zone. W. H. T.

557. Purification of Petroleum Oils. K. T. Steik, Assr. to Standard Oil Development Co. U.S.P. 1,791,941, 10.2.31. Appl., 30.8.28.

In this process the oil to be refined is treated with a preformed mixture of sulphur dioxide and sulphur trioxide which contains, in a particular instance cited, about 10 gm. of SO_3 per litre in solution. The reagent is agitated with the oil in a closed vessel at a temperature of —10° C. The oil may be treated with one or a number of batches of the reagent. After the treatment the mixture is allowed to settle and the oil and sludge layers separated. The oil is neutralised and finished with clay, and the sulphur dioxide evaporated from the sludge layer and liquefied for reuse. The oils thus refined possess high resistance to emulsification and are superior in this respect to oils refined by fuming sulphuric acid and subjected to the same finishing treatment.

R. W. L. C.

558. Treating Hydrocarbon Oils. W. M. Stratford, Assr. to Texas Co. U.S.P. 1,792,877, 17.2.31. Appl., 7.6.28.

A process is described of treating hydrocarbon oils to effect partial separation of unsaturated compounds of the same boiling point. It comprises the polymerisation of the unsaturated bodies to form higher boiling compounds by continuously contacting the oil in vapour form with a bed of adsorbent catalytic material. The adsorbed polymers are intermittently washed from the catalyst by a supply of a suitable solvent boiling within the approximate boiling range of kerosine. The temperature and pressure maintained in the catalyst are both increased as the activity of the catalyst tends to decrease.

W. H. T.

559. Converting Heavy Hydrocarbons into Lighter Ones. W. Landes. U.S.P. 1,792,912, 17.2.31. Appl., 10.7.26.

The apparatus described consists of a furnace with an upright chamber. The products of combustion flow upwardly through a vertical elongated expansion chamber placed inside the first chamber and having an arm projecting to one side between the ends of the expansion chamber. Hydrocarbon and steam are directed against the opposite walls of the expansion chamber through nozzles which extend through the entire length of this arm. The expansion chamber is also provided with an upper vapour outlet and a lower discharge means.

W. S. E. C.

560. Heating Oil and Superheating Steam. J. Primrose, Assr. to Foster Wheeler Corpn. U.S.P. 1,792,925, 17.2.31. Appl., 24.12.25.

The apparatus consists of a tubular oil heater with heating tubes absorbing radiant heat in a combustion chamber and a bank of tubes in a second chamber partly separated from the combustion chamber. The oil to be cracked is passed through a portion of the tube bank and the radiant heat-absorbing tubes at a velocity sufficient to avoid or greatly decrease any cracking of the oil. The oil-containing tubes are heated in the bank by passing heating gases into contact with tubes at a temperature and velocity such that the temperature of the gases leaving the tubes is about 1500° F. The steam is superheated in the remaining portion of the tube tank to a temperature of about 900° F. by contact with high temperature gases.

W. S. E. C.

See also Abstract No. 603.

Cracking.

561. Scientific Basis of the Cracking and Hydrogenation of Mineral Oils. V. Henri. *Rev. Pétrolifère*, 1931, (411) 202–217.

After a brief survey of the increased importance of cracking and a summary of the main groups into which the various cracking processes can be divided, three typical cases are outlined: (1) Aliphatic hydrocarbons with long straight chains in which rupture occurs between C—C in the middle of the molecule. (2) A shorter chain at a higher temperature, rupture occurring between C—C at the end of the molecule. (3) Benzene at 450—550° C. when polymerisation occurs with elimination of hydrogen.

A determination of the energy which binds the atoms together in a molecule is important. Thermo-chemical methods are too approximate, but recent work on absorption spectra has made it possible to calculate the energy necessary to break the bonds. For example, in aliphatic compounds the union C—C corresponds to 71 Kg.-cals, C—H 92 Kg.-cals., while C=C equals 125 Kg.-cals. For aromatic compounds C—C equals 112 Kg.-cals. and C—H 95—100 Kg.-cals. In the former case the strength of the bond varies at different points of the chain, the linking being weakest in the middle between C—C, so that at low temperatures the molecule splits into two halves, but at higher temperatures the end of the chain breaks. For aromatic hydrocarbons the C—H linking is weakest and rupture occurs at this point. This explains why naphthenes are difficult to crack and produce thick tars.

Investigation of the infra-red, ultra-violet and Raman spectra enable accurate measurements to be made of the dimensions, etc., of molecules, while recent progress permits the calculation of the "energy of activation"—the energy necessary to produce a certain reaction.

Sakhanow and Tilichow have shown that an increase of 10° C. doubles the rate of cracking.

A table is given of paraffins from pentane to hexadecane showing the usual constants together with decomposition temperatures and pressures.

A linear relation exists between the temperature at which a reaction occurs and the log of the speed of the reaction, *i.e.*, Log $V = A \times T$ or $V = e^{AT}$.

Molecules can only react if they have attained a certain "energy of activation" which, in the case of cracking has been found to be 25 Kg.-cals. per gram. mol. The temperature must therefore be raised until this value is reached.

The condition of reaction of a molecule of ethylene, for example, and a molecule of hydrogen is that the two molecules shall approach sufficiently close and remain in proximity for a sufficiently long time.

The "sphere of action" determines the minimum distance to which two molecules can approach during molecular collisions. An exact measure of this sphere of action can be obtained from a study of the viscosity of gases, while the kinetic theory enables a calculation to be made of the average time of contact between molecules. (For ethylene and hydrogen $= 10^{-13}$ secs.) For reaction to occur this time must not be less than 10^{-9} secs. The function of catalysts (particularly solids) is to prolong this time of contact. Gaseous molecules adhere to active points on the catalyst forming a mono-molecular layer and the time of adhesion is from 1/10 to 1 sec. For optimum conditions of reaction, these active points must be as near together as possible, and binary

and ternary mixtures, in which small atoms fill up the interstices of the network of the original catalyst and form new centres of attraction much closer together than the original, have been found to be most effective.

The potential energy of a molecule is a function of the distance between the atoms, and curves are given showing the relation between these two variables for the two pairs of radicles CH_3—CH_3 and CH_3—CH_2.

If, therefore, from a study of the spectra of any molecule, the distance between the atoms is known and also the distance between the atoms of another molecule the conditions necessary to produce reaction can be calculated.

These considerations apply equally to reactions between complex molecules as these appear to take place in intermediate stages which can be followed by examination of the spectra. Such methods also enable the energy of a dissociation of molecules to be determined. Figures are given for various molecules. For molecules containing more than one element, each of the various ways in which dissociation can occur corresponds to a particular energy. It is thus easily seen that in cracking a complicated compound such as mineral oil, many very different products can be obtained by altering the treatment.

The paper concludes with some 21 photographs of spectra of various compounds and mixtures and it is claimed that by these methods analyses can be made of mixtures which could not be done in any other way. C. C.

562. Cracking Gaining Steadily in Importance. E. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (38), 24.

The cracking of petroleum products was originally resorted to in order to increase the amount of gasoline available from crude. It was not at first popular with the motorists, but when improvements in the design of automobile engines led to the use of higher compression pressures it was found that such fuels were superior in anti-knock value. It is estimated that a third of the United States gasoline supply now consists of cracked motor fuel, compared with less than a fifth a few years ago. The increase in cracking has raised the total percentage of gasoline obtainable from the crude worked up in the U.S. from 32·43% in 1925 to 41·99% in 1930, resulting in an enormous saving in crude oil, which would otherwise have to be distilled to cope with the demand for motor spirit.

The yield of cracked benzine from various stocks is often over 70% and approaches 80% in some cases, while the quality is often of the order of 60 benzol equivalent or 75 octane number.

Modern cracking processes are sufficiently flexible to handle wide ranges of cracking stocks and to produce varying types of products. Valuable by-products are also produced at the same time. The coke, if not burnt under the stills or boilers in the refinery itself or in industrial power plants, may be used for the manufacture of electrodes or for metallurgical purposes, or may be briquetted. If there is no market for coke, the cracking plant may be operated under the residuum system with flashing, resulting in the production of marketable fuel. The pressure distillate bottoms may be used as domestic furnace oil or Diesel oil, or may be fractionated to give a good tractor fuel. The gases from the cracking plant may be utilised for enriching producer or water gas or worked up to propane and butane, which is sold in steel bottles under pressure for domestic fuel or for cutting metals. The synthesis of valuable chemical products from cracked gases is also increasing.

It is probable that the curtailing of crude oil production will result in the further development of cracking processes to provide the maximum amount of gasoline. C. L. G.

563. Cracking Paraffin Base Crude Oils. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (39), 38.

Owing to the relatively poor anti-knock value of straight-run gasolines prepared from Pennsylvanian crudes, experiments have been carried out on the cracking of Pennsylvanian kerosine (boiling range 322° F. to 649° F.) to produce cracked spirits of high volatility and anti-knock value, which can be used to improve the properties of the straight-run gasolines. In the past it was thought that cracking such distillates could not be economically carried out without resorting to very high temperatures and pressures, but the experiments reported show that the conversion can be carried out at temperatures ranging from 935° F. to 980° F. under a pressure of 350 lb., using the "flash" method of operation. The yields obtained ranged from 74·3% to 78·5% of gasoline, 4·7% to 11·3% of flashed residue, and 588 to 769 cu. ft. of gas per barrel of charging stock. The cracked gasoline obtained at a temperature of 980° F. had the following properties, as compared with straight-run Pennsylvanian gasoline :—

	I.B.P. °F.	20% dist. temp. °F.	F.B.P. °F.	% Aromatics.	% Unsaturateds.	% Naphthenes.	% Paraffins.
Straight-run ..	100	193	406	6·9	2·2	13·0	77·9
Cracked	90	182	384	29·3	33·0	14·2	23·5

The two spirits were tested in a "Series 30" ethyl knock-testing apparatus, determining the quantities of benzol which had to be added to a "zero" gasoline of low anti-knock value in order to equal the sample under test in anti-knock value. The octane numbers were calculated from the benzol values, using a pre-determined relationship.

The results were as follows :—

	% benzol equivalent.	Octane No.
Straight-run	33 ...	47
Cracked	61 ...	76

The cracked gasoline is thus better than the ethyl standard (74 octane number), whereas the straight-run gasoline requires the addition of 6·1 c.c. of T.E.L. to equal this standard.

The A.S.T.M. distillation, benzol equivalent and octane numbers of each 10% fraction of both straight-run and cracked gasolines are given in a series of tables, and demonstrate (*a*) that the heavy fractions are responsible for the poor anti-knock value of the straight-run gasoline, the octane numbers ranging from 77 for the first 10% to 14 for the fraction 90/97·5% (loss 2·5%); (*b*) the cracked gasoline fractions have a range of 86 to 64 octane number in descending order of volatility.

Details of the cracking method are given, also analyses of the various residues, which are shown to have low cold tests, viscosities and B.S. and W. contents, and are said to possess a high calorific value. The flash point (Cleveland) of the residues range from 125 to 165° F., and the percentage distilling at 572° F. from 34 to 51. R. A. E.

564. Cracking Process. F. C. V. Water, Assr. to Petroleum Laboratories Inc. U.S.P. 1,788,933, 13.1.31. Appl., 21.11.23. Renewed, 17.2.30.

Crude oil is heated in an ordinary topping still to vaporise the lighter fractions which are condensed; the heavier fractions in vapour form are passed through super-heating coils, long narrow pipes beneath the still, in which they are subjected to a temperature of 1000°–1300° F. The vapours then enter tubes of much greater diameter, in which the temperature is

maintained and in which they are subjected to a complete stabilisation, but without the deposition of carbon in appreciable quantities or further breaking down of the hydrocarbon molecules. From the stabilising tubes the vapours are taken to a condensing system. H. B. T.

565. Cracking Hydrocarbons. E. C. Herthel, Assr. to Sinclair Refining Co. U.S.P. 1,789,072, 13.1.31. Appl., 4.1.28.

A reflux column contains a series of baffle trays on to one of which in the lower part of the column is sprayed heavy oil charging stock, which may, if desired, have been heated by passing through a heat exchanger. In the upper part of the column are a number of spray nozzles, fitted over a corresponding number of trays, through which are introduced controlled quantities of a light oil refluxing medium. This may consist of a portion of the pressure distillate from the process and it should be substantially completely vaporised at the temperature obtaining in the column above the charging stock feed nozzle.

It is claimed that this method of working ensures accuracy of control, uniformity of operation, and maximum capacity. H. B. T.

566. Cracking Oils. F. Hofmann and C. Wulff. U.S.P. 1,791,562, 10.2.31. Appl., 26.11.28.

The use of boron halides is described as catalysts for cracking hydrocarbons. Addition or substitution products of these compounds may be employed. They possess great advantages over the chlorides of iron and aluminium, in that they are liquids or gases at atmospheric pressures and can thus be more readily brought into intimate contact with and also removed from the hydrocarbons than the solid metallic chlorides. When the cracking takes place in the presence of hydrogen, spirits of a saturated character and containing very little sulphur are produced. H. B. T.

567. Cracking Hydrocarbons. L. Kirschbraun, Assr. to Universal Oil Products Co. U.S.P. 1,791,566, 10.2.31. Appl., 1.9.20.

The plant described consists of a furnace with pipe still, a vapour chamber placed outside the furnace and a condensing system. The oil is maintained in the liquid phase in the pipe still to prevent precipitation and deposition of carbon in the tubes exposed to the heat of the furnace and to obtain satisfactory results it is found that the temperature of the oil as it leaves the pipe still must be kept within very narrow limits. To this end a pyrometer is installed in the transfer line between pipe still and vapour chamber and by means of thermostatic control the speed of the motor driving the oil feed pump is increased or decreased as the temperature tends to rise or fall. It is claimed that the oil temperature can be kept constant within 2° F., whereas by manual control a variation of 40°–50° is usual. H. B. T.

568. Converting Petroleum Oil. W. B. Howard, Assr. to Universal Oil Products Co. U.S.P. 1,791,622, 10.2.31. Original appl., 27.12.21. Divided 4.3.27.

The plant described comprises a horizontal cylindrical cracking still within which is an open-ended cylinder placed horizontally. In each end of this cylinder is a propeller driven by shafts passing through glands in the ends of the still. The charging stock is fed into the bottom of the still and the propellers keep the oil in rapid movement and prevent deposition of carbon on the bottom of the still.

The vapours evolved pass into a line from the top of the still, which enters the bottom of the still again and terminates in a nozzle under a small dome formed in the bottom of the hollow cylinder. From the top of the dome the main vapour line leads through a trap to a reflux column and fractionating system. The heavy condensate from the vapour line which flows back into the trap is led to a small reflux still, situated inside the main cracking still. The vapour line from the reflux still enters the main vapour line above the trap and excess oil in the reflux still is pumped to a third, cracking, still into which the reflux from the fractionating system also passes. It is here subjected to a more drastic cracking action and the vapours are taken into the main vapour line while the surplus oil is pumped into the charging line to the first still.

It is claimed that the character of the products is improved by passing the vapours from the main still backwards through the oil contained therein and also that there is advantage in returning the reflux for separate treatment, since it requires more severe cracking conditions which would bring about excessive carbon formation if applied to the raw charging stock.

H. B. T.

569. Cracking Oil. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. U.S.P. 1,791,617, 10.2.31. Appl., 25.4.21.

An improved apparatus for facilitating the cracking reaction in the vaporising zone in a cracking plant is described; also a means for distributing the carbon precipitated over the entire area of the expansion chamber.

Oil is heated in a coil in a furnace to a temperature of 700–800° F. and is maintained under a pressure of 20 lb./sq. in., and passed by means of a valve in a single passage to either of the opposite extremities of a horizontal vapour chamber. A means is provided for increasing the velocity of the oil as it reaches points near the entrances to the vapour chamber. The latter is connected to a condenser.

W. S. E. C.

570. Converting Hydrocarbon Oil. G. Egloff and J. C. Morrell, Assrs. to Universal Oil Products Co. U.S.P. 1,791,618, 10.2.31. Appl., 18.2.22.

Claims are made for a process for converting hydrocarbon oil in which the oil is raised to cracking temperature in a heating zone. The heated oil is allowed to vaporise and the vapours are subjected to dephlegmation and then subjected to a catalytic treatment. The treated vapours are condensed and collected together with the uncondensed gases. The latter are scrubbed, passed through a desulphuriser and then admitted to a flowing stream of dephlegmated vapours prior to the subjection of the dephlegmated vapours to the catalytic treatment.

W. H. T.

571. Converting Hydrocarbons. J. J. Jakowsky, Assr. to C. & C. Developing Co. U.S.P. 1,792,744, 17.2.31. Appl., 22.7.24.

The process of converting relatively heavy oils into lower boiling point products consists of vaporising the raw material under such conditions that a considerable part of the vapour is in a substantially saturated condition. The vapours, while in contact with a heated surface at a temperature above that of vaporisation, are subjected to the precipitating action of a high tension silent electrical discharge. This action tends to drive the non-gaseous part of the vapours towards the heated surface. The uncondensed products are conducted away for recovery of a desired lower boiling product therefrom.

W. H. T.

572. Treating Petroleum Oils. L. Huff, Assr. to Universal Oil Products Co. U.S.P. 1,793,443, 17.2.31. Appl., 17.5.22.

A process for cracking oils is described in which the oil is raised to a cracking temperature in a heating coil. The heated hydrocarbons enter the lower portion of a reaction zone from which vapours and liquid oil are removed separately. The return of the oil to the coil is prevented and the vapours undergo reflux condensation and the reflux condensate is returned for retreatment. The rate of withdrawal of unvaporised liquid from the reaction zone is controlled so as to prevent any accumulation of liquid in the bottom of the zone and the oil is maintained substantially above atmospheric pressure in the coil and the reaction zone. W. S. E. C.

573. Treating Hydrocarbon Oil with Metallic Halides. J. L. Cooley, Assr. to Standard Oil Co. U.S.P. 1,795,761, 10.3.31. Appl., 1.12.26.

This invention relates to a process and apparatus for the treatment of hydrocarbon oils by aluminium chloride. These two materials are mixed in a flowing stream at a temperature sufficient to cause them to react to form relatively low b. pt. oil. The stream is passed to a flash zone where low b. pt. vapours are separated from the liquid oil and aluminium chloride. A stream of the vapours and a separate stream of the oil and tar are passed to a conversion zone wherein the hot vapours are contacted with the oil-tar mixture. By this means, agitation is effected, and at the same time the mixture is maintained at a temperature suitable for effecting a decomposition reaction. W. H. T.

574. Treating Oils. C. B. Buerger, Assr. to Gulf Refining Co. U.S.P. 1,796,138, 10.3.31. Appl., 23.2.23.

The invention has for its object (amongst others) the cracking of oil in such a manner as to utilise to a high degree the heat of generated vapours for heating incoming oil. A body of oil is maintained under heat and pressure, the vapours being withdrawn under pressure, expanded and condensed. The charging stock is heated under pressure and passed into the main bulk of oil. Vapours from the heating means are allowed to mingle directly with those above the body of oil, while liquid oil is withdrawn from the latter and mixed with the incoming charging oil. W. H. T.

Hydrogenation.

575. Early Developments in Hydrogenation. V. M. Ipatiev. *Oil & Gas J.*, **29** (38), 30.

An account is given of the early history of the development of the hydrogenation process, commencing with the work of Berthelot 60 years ago on the decomposition of powdered coal by hydriodic acid under pressure. The work of early investigators and the theories developed on the mechanism of hydrogenation are outlined. A summary is given of the work being carried out by the Petrograd Academy of Science.

In brief it is believed that during hydrogenation extensive cracking takes place, this being increased by the presence of hydrogen under pressure; the cracked molecules are then hydrogenated. The use of catalysts lowers the temperature of reaction, which is usually above the dissociation temperature of many hydrogenated compounds, to one at which these compounds are stable.

A description is given of the Lernawerke plant near Merseburg (Germany) for the production of synthetic gasoline from the hydrogenation of coal. The brown coal used in this plant is very similar in chemical composition to montan wax, consisting chiefly of higher molecular hydrocarbons, alcohols and the corresponding saturated esters. The berginisation of waxy products is shown by N. Orlov to result in the formation of gasoline consisting chiefly of paraffin hydrocarbons. Rosins on berginisation yield products of polymethylene composition. The hydrogenation of brown coal may therefore be considered as consisting of two processes, low temperature carbonisation followed by hydrogenation of the products formed, the waxy compounds in the coal giving saturated hydrocarbons and the tars (which are similar to resins) giving polymethylenes. The phenols in the tar which are derived from the lignin and humic components of the coal are not much affected by hydrogen under pressure at high temperatures, simplification of structure chiefly only resulting—*i.e.*, removal of side chains.

In the presence of a catalyst, however, to lower the reaction temperature, phenols are rapidly reduced giving aromatic hydrocarbons. This is not to be confused with hydrogenation carried out with active catalysts where the benzene ring is hydrogenated, the hydroxyl groups remaining intact. A catalyst suitable for the latter type of hydrogen is alumina mixed with some metal oxides. Reduction of phenols can also be effected in an iron tin-lined or sulphurised tube, as shown by Fischer.

To obtain therefore the maximum yield from coal, the reaction should be carried out in two stages, the first stage producing a heavy oil without the presence of a catalyst, and the second stage converting the heavy oil and the phenols to lighter products under the effect of the catalyst. It is not sufficient to add the catalyst to the original coal, since it becomes occluded in the coal, and is also finally lost when removing the residues from the reaction chamber.

Recent work has also been carried out by the I.G. on the liquefaction of coal under elevated pressure at temperatures of 400° C. in suitable high-boiling solvents, a 60% yield being obtained. It is thought that a process of depolymerisation rather than simple extraction is involved.

The berginisation of hard coal is not so easily effected, owing to the inert nature of the fusain. An interesting result of light berginisation is to render the material easy to coke. According to Lander, this may give a means of producing good coking coal from anthracite.

It is pointed out by the author that the production of oil from coal under pressure at high temperatures resembles the natural production of petroleum in nature, although lower temperatures are usually experienced in the earth. Magnetic fields may also increase the yield of oil from coal in the earth.

C. L. G.

576. Hydrogenation of Carbonaceous Bodies in the Presence of Catalysts. Chemical Reactions, Ltd. E.P. 341,560, 22.1.31. Appl., 22.10.29.

The invention is especially concerned with a preliminary treatment of tars, mineral oils, etc., to remove ash and other more or less insoluble substances which have an injurious effect on the catalyst. Even small quantities of these substances are liable to separate out during the hydrogenation process on to the surface of the catalyst, so that the activity of the latter is greatly reduced, and it has been found that when the catalyst has been thus covered it cannot in practice be economically regenerated. Injurious constituents are therefore removed in a separate vessel, by passing the raw material under pressure of hydrogen over an inert filling material on which the injurious materials are deposited. Pumice, Raschig rings, clay or porcelain filling materials, which are incapable of exerting a catalytic action, may be used.

If the starting material contains asphaltic matter the latter separates along with the ash, and usually in such amounts that the ash constituents flow away from the inert filling, in solution or suspension, and can be drawn off at the bottom of the vessel. Thus the separation of the ash is not interrupted and the filling material does not need to be renewed. If the asphaltic material is small in the initial feed, asphaltic substances can be added to provide sufficient material to cause the ash to flow off.

No definite temperature can be stated as this will depend on the raw materials, but the aim is, wherever possible, to adjust the process so that the conditions in the hydrogenating vessel are reproduced in the separating vessel, in order to eliminate any material which would become insoluble under these conditions. R. P.

577. Destructive Hydrogenation of Coal and Oils. J. Crow, J. Gordon, and Imperial Chemical Industries Ltd. E.P. 341,676, 22.1.31. Appl., 17.12.29.

Hydrogen which has been used for destructive hyrogenation and which is contaminated with hydrocarbons and possibly nitrogen, is purified by washing with a suitable oil under pressure at a temperature below 0° C. The dissolved gases may be recovered from the solvent oil by a step-wise reduction of the pressure. When it is desired to remove nitrogen from the system this may be done simultaneously with the recovery of the bulk of the hydrogen, methane, and ethane, by adjusting the pressure or temperature by which the release of gas from the solvent is brought about.

In another modification of the invention the crude gases are first washed with sufficient solvent to dissolve the bulk of the ethane, and the residual gas is further treated with quantities of the same or a different solvent to remove principally methane.

In the case of a circulating system where nitrogen tends to accumulate, ethane is first removed by a limited amount of solvent; then, using the calculated quantity of solvent, the required amount of nitrogen may be removed. Care must be taken that the solvent is not cooled below solidification point. In some cases the condensate produced in the process is suitable. Petrol consisting principally of paraffins may be used at a very low temperature, but aromatic and naphthenic petrol cannot be cooled to the same extent.

The paraffin gases removed by this method may be utilised for the production of more hydrogen by known methods. R. P.

See also Abstracts Nos. 502, 561, 609.

Chemistry of Petroleum.

578. Properties of Petroleum Hydrocarbons. W. R. Nelson. *Petr. Eng.*, 1931, **2** (5), 97.

Tables are given showing the physical constants of the more important hydrocarbons occurring in petroleum, these being divided into Diolefines, Aromatics, Olefines, Naphthenes, Paraffins, Acetylenes and Iso-Paraffins. The data given include mol. wt., gravity, b. pt., m. pt., latent heat, heat of combustion and (in a few cases) heat of fusion and entropy.

Extensive references are given to the papers from which the data have been taken, together with indications of the method of calculation of the various constants. C. L. G.

579. Occurrence of Higher Fatty Acids in Mineral Oil Distillates. E. Holzmann and St. v. Pilat. *Brennstoff Chem.*, 1931, **12** (3), 41–42.

In the previous research on the phenols of petroleum it was found that a mixture of naphthenic acid crystals separated from the petroleum distillates of Boryslaw crude oil, consisting of higher fatty acids (*cf. J.Inst. Pet. Tech. Abstr. No.* 1050, 1930).

The initial substance utilised in the present research was a spindle-oil distillate from Boryslaw petroleum of the following properties: D_{15} 0·901, visc. 5·58° E. at 20° C., solidification point + 0·5° C., flash point 173° C. Twenty-nine and a-half kgms. of this distillate were mixed vigorously in an agitator with 1,000 litres of soda lye (7° Bé) for 6 hours at 50·55° C. After 24 hours the lye was run off and dilute sulphuric acid added to give an acid reaction; 40·5 kgms. of crude naphthenic acids and phenols separated. The water-free acid mixture was distilled in the same apparatus at 12–15 mms. Hg; the temperature in the distillation vessel was 240–245° C.; that of the dephlegmator 235–240°.

The distillate was separated into the following components:—

Product.	Gms.	% calculated on distillate.	% of crude naphthene-phenol mixture.	% of original oil.
Acids	1149	18·6	9·11	0·0125
Phenols	645	10·4	5·03	0·0069
Hydrocarbons	4290	—	—	—
Loss	111	—	—	—

A crystalline precipitate separated on cooling and after centrifuging from the acid mixture freed from phenols and hydrocarbons. The phenols were extracted by conversion into the lithium salts. The crystalline precipitate was crystallised from methyl alcohol and recrystallised from acetone. Pure white crystals of m.p. 70·5° C. were obtained, which contained arachidic acid. $C_{20}H_{40}O_2$. W. S. E. C.

580. Physiochemical Properties of Acid Clay. V. Decolorisation of Petroleums by Adsorption. (1), (2), (3). K. Kobayashi and K. Yamamata. *J.Soc. Chem. Ind.* Japan, 1930, 33. Suppl. binding, No. 11, 428–431.

The Freundlich adsorption formula $x/m = aC^{\frac{1}{n}}$, in which x is the quantity of dissolved substance adsorbed, m is the quantity of adsorbent, and a and n are constants, applies during the decolourising of various petroleum distillates with Japanese acid clay. Decolourisation takes place mainly by adsorption. The Freundlich formula applies also to the action of fullers earth, Florida earth, German activated clay, silica gel, and Norit carbon, and holds true with a benzol solution of oil yellow and kerosine solution of resins extracted from machine oil. The orders of decolourising action of acid clays, etc., upon different series of crude petroleums, their distillates, etc., are parallel to each other. Decolourising powers of acid clays upon a water solution of methylene blue or a benzol or kerosine solution of oil-soluble dyes are not parallel to the case of petroleum distillates. An asphalt-kerosine solution is adopted for evaluating acid clay. The decolourising power of acid clay on petroleum is increased slightly by a temperature rise from 15–95°. Acid clay dried at 125–50° has the greatest decolourising action on petroleum. W. S. E. C.

581. Behaviour of Certain Thiophanes in Heptane and Naphtha Solutions. R. W. Bost and W. M. Conn. *Ind. Eng. Chem.*, 1931, **23**, 93–95.

In 1906 Mabery and Quayle isolated a series of sulphur compounds from Canadian petroleum having the general formula $C_nH_{2n}S$ to which the name "thiophanes" was given. Thiery isolated two members, tetramethylene and pentamethylene sulphides, from the acid sludge obtained in the refining of Persian petroleum. The present paper gives results indicating the behaviour of tetramethylene and pentamethylene sulphides with various reagents in pure *n*-heptane and three naphthas. Alongside these experiments were conducted similar ones in which ethyl sulphide (representing alkyl sulphides to a certain extent) and thiophane (representing thiophenes) were used. The concentration of sulphur used in the work was 1·8%, and the reagents employed were mercuric chloride, mercuric iodide, 30% hydrogen peroxide in glacial acetic acid, nitric acid (S.G. 1·42), potassium permanganate, bromine and methyl iodide. The results are detailed in the text and conveniently listed in tabular form.

With mercuric chloride and mercuric iodide, precipitates were obtained in all cases except thiophene, whether *n*-heptane or the naphthas were the solvents. With nitric acid, sulphones resulted, except from thiophene, when *n*-heptane was the solvent, but no definite products could be isolated when the sulphur compounds were dissolved in the naphthas.

The work with permanganate was carried out after the method of Mabery and Quayle, and with all the sulphides when *n*-heptane was used, the corresponding sulphones were obtained. When the naphthas were dissolved, the reaction products were less pure and needed several recrystallisations. Variations in purity of these products also depended on the particular naphtha. The action of hydrogen peroxide gave results similar to those where permanganate was used. Methyl iodide gave iodomethylates, thiophene excepted, the purity of which was again dependent on the solvent.

Bromine gave unstable dibromo addition products, thiophene again being an exception.

In brief, therefore, the behaviour of these compounds with the reagents is modified by the solvent—an observation which falls in line with previous work on other sulphur compounds. Thirteen references are appended.

C. I. K.

582. Action of Inorganic Refining Reagents on Alkyl Sulphides in Naphtha. P. Borgstrom and J. C. McIntire. *Ind. Eng. Chem.*, 1931, **23**, 321-323.

The effect of mercuric acetate and mercuric chloride solutions, solid mercurous nitrate and silica gel on fourteen alkyl sulphides in naphthas has been studied. The alkyl sulphides chosen were ethyl, *n*-propyl, *iso*-propyl, allyl, *n*-butyl, *iso*-butyl, *sec*-butyl, *n*-amyl, *iso*-amyl, *sec*-amyl, *sec*-hexyl, *n*-hectyl, *sec*-octyl, benzyl. Four different naphthas were used.

The results with mercuric acetate solution showed that the lower sulphides are more readily removed than the higher, while isobutyl and isoamyl sulphides were removed about the same as the corresponding normal sulphides. The *sec*-propyl and *sec*-butyl sulphides were removed more than the corresponding normals, but as the length of the carbon chain increased the effect was very small. Similar results were obtained with mercuric chloride solutions with an exception in the case of allyl sulphide, of which very little was removed by this treatment. In short, alkyl sulphides having two or three carbons in the chain are almost completely removed, but those with five or more carbons are but slightly touched by these two solutions.

Solid mercurous nitrate proved to be very efficient in removing the lower sulphides and some of the higher normal sulphides, but was not very effective with the higher secondary sulphides. Owing to their high boiling points it is questionable whether these higher secondary sulphides would be present in ordinary gasoline.

Treatment with silica gel showed that there was more of the *sec*-propyl and *sec*-butyl removed than the corresponding primaries, but with the amyl there was a very small difference. The removal is obviously dependent upon the length of the chain and there is no sharp break in the curve.

A naphtha containing the sulphides mentioned was also treated with various inorganic reagents. Cupric sulphide removed an appreciable amount of alkyl sulphide, but zinc sulphide did not. Aluminium oxide gave promising results, while zinc, stannous and cupric chlorides were quite effective.

W. D. S.

583. Studies in the Naphthalene Series. Part III. The Preparation of 3-Amino-2-naphthyl Methyl Ether. Part IV. Preparation and Properties of 2-Naphthol-3-Sulphonic Acid. G. B. Jambuserwala, S. Holt and F. A. Mason. *J.C.S.*, 1931, 134, 373-381.

III. Methods previously described for the synthesis of 3-amino-2-naphthyl methyl ether give unsatisfactory yields and two new methods are described.

(1) 2-hydroxy-3-naphthoic acid→methyl ether→acid chloride→amide→3-amino-2-naphthyl methyl ether. The Hofmann reaction for the conversion of the acid amide into 3-amino-2-naphthyl methyl ether did not give a very good yield.

(2) 2-Hydroxy-3-naphthoic acid→methyl ether→ethyl ester→hydrazide→azide→carbamate→3-amino-2-naphthyl methyl ether. This method gives very good yield.

IV. 3-Amimo-2-naphthyl methyl ether (see preceding abstract) was diazotised, the solution treated with an excess of sulphur dioxide and chemically prepared copper powder added. When the reaction was complete the solution was filtered and the sulphinic acid extracted from the filter cake with 30% aqueous potassium carbonate. The sulphinic acid was oxidised in suspension in acetone with finely powdered potassium permanganate, an excellent yield of the sulphonic acid being obtained. A. J. G.

584. Kinetics of the Polymerisation of Ethylene at Pressures above one Atmosphere. R. N. Peaco. *J. Amer. Chem. Soc.*, 1931, **52**, 613.

Previous workers have shown that ethylene in glass at 400—600° C. and 1 atm. initial pressure polymerises to produce mono-olefines. Under these conditions polymerisation is slow and limited. Secondary reactions readily occur. Indications suggest that polymerisation is a second order reaction and decomposition a first order reaction. Increase of pressure favours the former.

Using a copper bomb of 250 c.c. capacity in the temperature range 350—500° C. and initial pressures of 2.5, 5 and 10 atm. it was shown that as much as 50% of the ethylene used, may be polymerised without the formation of more than 1 or 2% of hydrogen and methane. Under suitable conditions the only change was the production of butylene. Although no direct tests were made it was thought that the polymerisation reaction was homogeneous.

Velocity constants calculated on the assumption of a second order reaction agreed reasonably well, but an abnormally low temperature coefficient was found. A full consideration of this aspect of the subject is given.

E. N. H.

585. Pyrolysis of Propylene. C. D. Hurd and R. N. Meinert. *J. Amer. Chem. Soc.*, 1930, 51, 4978.

Preliminary experiments indicated that propylene was not appreciably changed at temperatures below 525° C. in pyrex glass apparatus with a hot contact time of 50 sec. Even at 600° C. the change was only 4% with a contact time of 30 sec.

It was concluded that the reaction in pyrex was predominantly homogeneous and unimolecular since dilution with nitrogen, or increased surface had a negligible effect on the proportion of propylene decomposed in a given time.

As products, methane, ethylene, hydrogen and paraffins higher than methane (chiefly ethane) were found in the ratio 3 : 3 : 1 : 1. In pyrex, or in quartz, one-fifth to one-fourth of the propylene used was converted into liquids. Benzene and toluene were identified and naphthalene and phenanthrene were indicated.

Using a mixture of equal parts of hydrogen and propylene it was found that the amounts of ethylene and methane produced were greatly increased. Little or no propane was formed. This indicates a scission :—

$$CH_3—CH=CH_2+H_2 \rightarrow CH_4+CH_2=CH_2$$

With propylene in monel metal the almost exclusive change was a decomposition into hydrogen and a sooty form of carbon. Much more propylene decomposed in monel metal at 350° C. than in pyrex at 650° C. with the same contact period. The reaction in monel was autocatalytic; as carbon deposited the decomposition became more rapid. In appearance and quantity this carbon differed from that obtained with pyrex. The pyrolysis started about 350° C. and at 375° C. with a contact time of about ½ min. it was practically complete. About 1 litre of hydrogen was produced from every litre of propylene at 375°, while at 650° C. 1.7 litres were obtained.

The relative stability of propylene, isobutylene and allene was considered. Propylene and isobutylene closely resembled one another. Pyrolysis of allene was quite different.

The effect of shorter contact times and higher temperatures was considered by using a quartz tube at 950° C. and a contact time of ½ sec. Under these conditions practically the same results were obtained as by pyrolysis in pyrex at 700° C. with a contact time of 53 sec.

Full experimental details are given. E. N. H.

586. Berginisation of Anthracene. N. A. Orlov and N. D. Likhachev. *Ber.*, 1930, 63 B., 2179-85; *Chem. Abs.*, 1931, **25**, (4), 695.

The study of the behaviour of condensed ring systems towards H_2 under pressure at high temperatures has been extended to anthracene. From the results of its destructive distillation with aluminium chloride and the behaviour of phenanthrene it was anticipated that anthracene would first form tetracene which at the high temperature would undergo rupture of the tetramethylene ring with the formation of $C_{18}H_8$ and its homologues. The latter would again be hydrogenated to tetrahydrides and decomposed, finally yielding aromatic hydrocarbons. Experimental evidence confirmed these expectations, for in the mixture of liquid hydrocarbons obtained by the berginisation of anthracene with sodium as catalyst, the following were identified: aromatic hydrocarbon (with traces of saturated hydrocarbons), tetralin, $C_{18}H_8$, β-$C_{18}H_7Me$, β-$C_{18}H_7Et$, and the tetrahydrides of the last two compounds. The non-picrate forming part of the hydrocarbons was investigated by dehydrogenation with Se or S. Se only gave moderate results, but with S, the dehydrogenation apparently proceeded regularly,

the greater part of the hydrocarbons changing into an asphalt-like substance and only small quantities of alkylnaphthalenes being isolated. No paraffins or olefines were present in the materials subjected to dehydrogenation, the anomalous results being ascribed to the presence of five membered ring hydrocarbons which may be formed by isomerisation of the tetralins to hydrindenes. In order to verify the possibility of the reaction tetralin→methylhydrindene, pure tetralin was heated under pressure and by fractionation of the product, several tetralin-free fractions were obtained and separated into aromatics and methylhydrindene. W. H. T.

587. Reactivity of Halogens in the Halogeno-derivatives of Cyclohexane. B. V. Tronov and L. V. Ladigina. *Ber.*, 1930, **36** [B], 3060-3067.

The rates of reaction of cyclohexyl chloride, bromide, and iodide and of a sec-hexyl iodide (about 70% of β and 30% of 7-iodohexane from mannitol with hydriodic acid, iodine, and phosphorus) and the corresponding bromide with sodium methoxide, pyridine and piperidine, respectively, have been measured at 17–19°, the ionised halogen being determined gravimetrically. The halogeno-derivatives of cyclohexane react less readily than the aliphatic halides with amines and potassium iodide, but very energetically with alkoxides. In their reactivity towards amines they very greatly excel the nuclear-substituted aromatic halogeno-compounds. The closure of the six-membered ring does not invariably diminish the readiness with which halogen is lost, but causes a similarity between the halogenated cyclohexanes and the tertiary alkyl halides. In general, the halogen is not replaced but removed mainly, and often exclusively, as hydrogen halide with the production of cyclohexene. An electronic interpretation of the course of the reaction is given. W. S. E. C.

See also Abstracts Nos. 561, 592.

Analysis and Testing.

588. Research on Paraffins, Stearin and Similar Substances. F. Dangl. *Petr. Zeit*, 1931, **27** (8), 142-144.

An accurate method for the determination of the melting and solidification points of paraffins, stearin and similar substances is described in which a microscope is used. The smallest quantity of the substances is necessary and this is examined in polarised light. An increase in the intensity of the field of light is observed in the solid state and the structure of the solid is very significant. The field of light remains dark when the solid melts.

In carrying out m.p. determinations under the microscope an adjustable electric thermo-element or resistance thermometer is used for temperature measurements; the m.p. can be determined accurately by measuring the intensity of the field of light by means of a photoelectric cell. A curve is drawn showing the relationship between the temperature and intensity of light, the sharp break in the curve indicating the melting-point or solidification point.

It is possible to estimate the relative quantities of the constituents present in a product, which melt, at different temperatures by means of a "Netz Okular Micrometer" which is placed in the eyepiece of the microscope. Greatly magnified photographs of samples of commercial paraffin which possesses a spongy structure, of beeswax, stearin, and candle wax are given.

W. S. E. C.

§89. Determining Gum Contents of Gasolines. O. C. Bridgeman and E. W. Aldrich, *Oil and Gas J.*, 1931, **29** (37), 42.

The above paper presented by representatives of the Bureau of Standards at the annual meeting of the S.A.E., gives a resumé of the more important methods for determining gum contents of gasolines and of the results of an extensive investigation into the most satisfactory method of carrying out this test. The problem of gum in gasoline divides itself naturally into three phases:—1. The development of a method for the determination of the gum content of gasolines at the time of test. 2. The development of a method for predicting the gum content of gasolines stored for a given time under special conditions. 3. The correlation of data obtained by the methods developed in the first two phases with the results of engine tests.

The essential feature of a method designed to give the gum content at the time of test is that the evaporation should be as rapid as possible to prevent the formation of gum during the test. The chief methods that have been used as are follows:—(*a*) Copper Dish Test (Method 530.1 Bureau of Mines Technical Paper 323B).; (*b*) Steam Oven Method—Glass Dish (Bureau of Mines Rept. of Investigations No. 26F6, 1925); (*c*) Air jet Method (J. Soc. Aut. Eng., 1930, 1, 31).

Full descriptions of the above methods together with the necessary precautions to be taken are given.

Comparative results by the above methods indicate that the copper dish method gives the highest result, while in some cases the glass dish and air jet methods give similar results and in others the air jet values are distinctly lower than the glass dish values. It is felt that the method giving the lowest results consistently is the most significant method, while high results are very easily obtained owing to the difficulty of drying gums to constant weight.

The results of a comprehensive series of tests on the above three methods are given in a table. It is shown that the air jet method gives more reproducible results than the glass dish, while the copper dish method gives the least reproducible results. Also the air jet method gives a fairly definite relationship to the gum content of a gasoline compared with that of the same gasoline blended with an equal volume of a gum-free gasoline. The other two methods, particularly the copper dish method, give results for 50/50 blends much less than half those given for 100% gumming gasoline.

It was found that to bring the gum formed to constant weight the following drying periods were necessary:—Copper dish, 5 to 29 hours; Glass dish, 5 to 22 hours; Air jet, 3 to 16 hours.

Investigations of the air-jet method showed that rates of flow of air below 300 ml. per sec. (through a jet 2.5 cm. above dish) give higher results, while variation of flow between 300 and 900 ml. per sec. (representing the safe upper limit of evaporation) has no effect. The use of CO_2 or nitrogen is cumbersome and gives higher results than the use of air for evaporation. Further work on the use of nitrogen is, however, in hand. While the air jet method gives the lowest gum values it does not entirely satisfy the 50% relation, so that gum may be formed during the test. An exact determination of the gum present could be made if a suitable solvent were found, but so far such a solvent has not been discovered. Further work is being carried out to study the effect of the variables (*a*) time of evaporation (*b*) temperature (*c*) atmosphere surrounding the gasoline during evaporation. It is hoped by varying the rate of evaporation to produce a curve of the relationship of gum content to rate of evaporation, and to extrapolate the values to find that at zero time, indicating instantaneous evaporation. Similar tests with varying volumes have been carried out, and a curve drawn showing

the change in apparent gum content of these gasolines with the volume evaporated. From these curves the gum formation at zero volume and time can be extrapolated.

So far there are no engine results available to determine which method gives results most in line with actual practice. In view of the expensive nature of such tests and the varying factors involved, it is felt that a laboratory flow method should be devised to simulate engine manifold condition.

References are given to the work of previous investigators on the subject.

C. L. G.

590. Spontaneous Ignition of Fuel Oils. (Determination of). W. Dunkerley. *Petr. Times*, 1931, **25**, 389-392.

Existing methods for determination of spontaneous ignition temperatures criticised under following headings :—(1) Discrepancies between temperatures of oxygen and ignition chamber. (2) Use of solid drop of test oil involves errors due to loss of lighter constituents. (3) Determinations can be carried out at atmospheric pressure only. (4) Dissimilarity to cylinder head conditions.

Improved apparatus is suggested in which a silica tube is electrically heated and so mounted that its interior may be at other than atmospheric pressure. A plate glass window allows of observation of the interior. Oil partially preheated by exhaust is introduced through injector type sprayer and oxygen through system which brings it to temperature of test before entering the ignition zone.

H. G.

591. Vapour Pressure-Temperature Relationships of Petroleum Hydrocarbon Fractions. S. D. Turner and J. E. Pollock. Trans. Am. Inst. Chem. Eng., New Orleans Meeting 1930, 15 pp.

In refinery practice it is frequently necessary to extrapolate a vapour pressure from the temperature at which it was determined to a working temperature or from one working temperature to another. It has been customary to perform such an operation on a vapour-pressure chart of the normal paraffin hydrocarbons. Unsatisfactory results in design work based on the application of this method, together with fragmentary data from various sources, led to the belief that such an extrapolation was at times not even approximately correct. While the investigation has not covered nearly the whole range of petroleum fractions, or of temperatures and pressures of interest to the engineer, it is felt that two phases of the work will be of interest; in the first case a very useful correlation is reached, and in the second a new laboratory method of investigation is developed. The paper treats of the theoretical considerations involved, the correlation of gasoline data, the vapour pressures determined by their experimental method, which is outlined, and the method and results are considered in a critical manner.

W. S. E. C.

592. Vapour Pressures of some Hydrocarbons. E. G. Linder. *J. Physical Chem.*, 1931, **35**, 531.

A series of determinations of the vapour pressures of a number of hydrocarbons has been made in the neighbourhood of 0°C. and figures are given.

The method used was modification of that due to Ramsay and Young. The apparatus consisted essentially of a glass chamber containing a thermometer on the bulb of which were a few layers of cheesecloth saturated in the hydrocarbon. This was connected to a liquid air trap and thence to a 3-litre flask containing air. Connections to vacuum pump and McLeod gauge were made from the flask. The apparatus was first pumped out until the thermometer cooled by the evaporation of the hydrocarbon registered a temperature near

0°C. The stopcock to the pump was then closed, and sufficient time allowed for equilibrium to be reached when a pressure reading is taken. The liquid air trap prevents the contamination of the air in the 3-litre flask, so that the McLeod gauge was functioning on pure air. P. D.

593. Specific Heat of Gases at High Pressures II. Method and Apparatus at High Temperatures. N. W. Krase and B. H. Mackey. *J. Amer. Chem. Soc.*, 1930, **52**, 5111.

In the first paper (*J. A. C. S.*, 1930, **52**, 108), the apparatus was described for the determination of the specific heat at ordinary temperature and at pressures up to 1000 atm. The present paper describes the modified apparatus which may be used up to 350 °C., and over the same range of pressures.

The apparatus is a constant flow adiabatic calorimeter. The gas is delivered to the calorimeter at the desired pressure and temperature, the preheating having been carried out in a thermostat which consisted of 3.5 m. of 6.5 mm. by 1.5 mm. steel tubing round which is cast a cylindrical block of copper. The heating is electrical, and temperature fluctuations are observed on a Beckman thermometer. The temperature is controlled manually, and can be held constant to 0.01° C.

The gas is preheated to a temperature about 5° C. below that of calorimeter, and passed to it through a well-lagged tube. The calorimeter consists of a copper cylinder 7.5 cm. long, 6.4 cm. diam. in which is embedded the coiled tubing through which the gas flows, and a thermometer. The block also contains an electric heater. The inlet and outlet tubes are vertical and the block is placed at the bottom of a Dewar flask 29 cm. long. The Dewar flask is enclosed in thick copper case which is electrically wound and can be maintained at the same temperature as the calorimeter. The inlet and outlet temperature of the gas is measured by two thermocouples, the wires of which pass down the vertical inlet and outlet tubes. The wires pass into the heated zone through bakelite compression seals which are 13 cm. from the top of the copper jacket, and may be easily kept cool. There are five equally spaced couples on each vertical limb, so that the temperature gradient may easily be determined and the heat loss along these tubes calculated.

In order to determine the outlet temperature of the gas, preliminary experiments were performed in which it was shown that the heat transfer in the calorimeter is nearly perfect, the outlet temperature rising to within 0.06° C. of the calorimeter temperature.

Rates of gas flow of 0.7 to 1.5 mol. min. were used.

A typical set of results for nitrogen at 500 lbs. pressure are given.

P. D.

594. Estimation of Unsaturated Hydrocarbons in Gases (III.). H. S. Davis, G. S. Crandall, and W. E. Higbee, Junr. *Ind. Eng. Chem.*, 1931, **3** (1), 108-110.

This deals with an experimental investigation of the factors affecting the completeness of the titration of unsaturated hydrocarbons by bromine using the bromide-bromate method of Francis.

A method has been evolved whereby ethylene and acetylene and some of their higher homologues can be quantitatively estimated by titration and a measure of success has been achieved in the analysis of synthetic mixtures of gaseous olefines and acetylenes.

It is thought that the presence of oxygen may be mainly responsible for irregularities encountered in halogen titrations of acetylenes by other methods.

H. B. T.

595. Examination of Tar and Mineral Insecticides. H. Martin. *J. S. C. I.*, 1931, **50**, 91T-94T.

Mineral oil preparations and tar-distillate washes are in general used as ovicides; little appears to be known, however, regarding the character of the actual mineral oil or tar used in these emulsions.

To determine the mineral oil, 50 gms. of emulsion, diluted with water are shaken with 10% sodium hydroxide. Extract with methylated ether, adding a small amount of sodium chloride to hasten the breaking if necessary. The oil is obtained from the ether extract after washing, etc., as usual. Phenols if present may be determined from the sodium hydroxide solution.

The mineral oil for physical and chemical tests is separated from the emulsion by shaking with saturated salt solution and heating on a water bath. To evaluate mineral oils for insecticidal use, the following characteristics are suggested: Volatility, content of unsaturated and aromatic hydrocarbons, ease of oxidation, presence of sulphur compounds.

The conclusion reached by Gray and de Ong (*Ind. Eng. Chem.* 1926, **18**, 175) that ingredients of petroleum oils which cause injury to plant foliage are removable by sulphuric acid, has been confirmed in practice.

Tar distillate washes.—The active ovicide appears to be high boiling neutral oils, the presence of tar acid decreasing the effectiveness. Neutral oils and tar bases can be separated as above. The latter can be removed and estimated by washing the ether layer with hydrochloric acid. The neutral oil content is obtained from the ether layer.

Tar acids are determined from the caustic soda washings after precipitating resins, soaps, etc., with barium hydroxide by acidifying and extracting with ether.

It is suggested that a volatility test is preferable to a distillation test both for tar and mineral oils, to ascertain the permanence of the oil deposit remaining after spraying. W. A. W.

596. How to Obtain Water Samples for Analysis. H. S. Peters, *Oil & Gas J.*, 1931, **29** (40), 130, 131.

Carefully cleaned 1 gal. glass stoppered bottles should be used for sampling, the stoppers tied down with cloth. The bottles should be thoroughly rinsed with the water to be sampled, filled to overflowing, emptied and refilled, leaving sufficient space to allow for expansion, and a piece of cloth tied firmly over the neck to hold stopper in place. It is important to avoid (1) scouring interior with the fingers or with cloth (2) using sealing wax on stopper, (3) using stoneware jugs, as they are difficult to clean and some of the salts used for glazing may remain. A quantity of water should be run to waste before sampling, when drawing from taps or pumps. Samples from a lake or stream should be taken well out from shore, and the bottle sunk to mid-depth before opening the stopper to avoid surface scum and bottom mud. Complete information, including date, location, nature of surrounding country and buildings and nature of soil should be given for the guidance of the analyst. Since water is subject to rapid changes the analysis should be made as soon as possible. If the sample has to be sent away or stored, it should be closely packed in ice. R. A. E.

See also Abstracts Nos. 510, 512, 598.

Anti-Detonation.

597. Detonation Characteristics of Some Paraffin Hydrocarbons. W. G. Lovell, J. M. Campbell, and T. A. Boyd. *Ind. Eng. Chem.*, 1931, **23**, 26–29.

Previous work has shown different classes of compounds to have widely different knock tendencies, and this applies also to isomers (*cf.* octanes). This paper extends this knowledge to include twenty-seven paraffin hydrocarbons, and the data presented are believed to form a picture of the relationship between structure and detonation characteristics for paraffin hydrocarbons boiling within the gasoline range. The knocking tendencies of these substantially pure hydrocarbons were compared by the bouncing-pin method in admixture with a reference gasoline and expressed in terms of the anti-knock effect of aniline. A positive aniline equivalent indicates the compound knocks less than the reference gasoline, and represents the amount of aniline, expressed as the number of centigram mols (*i.e.*, gram-mols × 100) per litre, which must be added to the reference fuel to produce a mixture that is equivalent in tendency to knock to 1-molar solution of the compound in the reference gasoline (*i.e.*, 1 gram mol of compound made up to 1 litre with reference gasoline). A negative equivalent indicates that the compound knocks more than the reference gasoline and represents the amount of aniline expressed as centigram-mols per litre which must be added to the molar solution of the compounds in the reference gasoline to make it equivalent in knocking tendency to the reference fuel.

The accuracy is within $\pm 1\%$ of a unit of aniline equivalent for compounds upon which two or more determinations were made. The magnitude of the scale of knock rating is shown by the following. Normal heptane of −14 aniline equivalent and 2, 2, 4-trimethylpentane of + 16 aniline equivalent knock at compression ratios of 2·75 : 1, and 6·9 : 1 respectively. The data do not permit of direct translation of values into terms of other scales, but the method used gives a measure of the partial knock effect of each compound at concentrations which are comparable with those in which such a compound might occur in commercial gasolines. However, it makes apparent the consistent relationships between molecular structure and knock rating. The following aniline equivalents are given: *n*-pentane, 1; 2-methylbutane, 9; 2, 2-dimethylpropane, 15; *n*-hexane, −6; 2-methyl pentane, 4; 3-methylpentane, 8; 2, 2-dimethylbutane, 13; 2, 3-dimethylbutane, 19; *n*-heptane, −14; 2-methylhexane, 0; 3, methylhexane, 3; 2, 2-dimethylpentane, 8; 2, 3-dimethylpentane, 12; 2, 4-dimethylpentane, 8; 3, 3-dimethylpentane, 13; 2, 2, 3-trimethylbutane, 19; 3-ethylpentane, 4; n-octane, −21; 2, 5-dimethylhexane, 5; 2, 2, 4-trimethylpentane, 16; 2, 2, 3-trimethylpentane, 17; 2, 2, 3, 3-tetramethylbutane, 26; *n*-nonane, −28; 2, 6-dimethylheptane, −6; *n*-decane, −30; 2, 7-dimethyloctane, −10; 3, 3, 4, 4-tetramethylhexane, 29.

The data were plotted and relationships shown are discussed under four headings, which in brief indicate that in an homologous series knocking tendency increases with increasing length of the carbon chain. In an isomeric series, the knocking tendency usually decreases as the number of side chains is increased. The successive introduction of methyl groups into a carbon chain of given length increases the tendency to knock by substantially a constant increment for each methyl group added. C. I. K.

598. Oil Consumption and Anti-knock Rating. H. R. Stacey. *Oil & Gas. J.*, 1931, **29** (38), 26.

Tests have been carried out in an Ethyl Gasoline Corp. water-cooled engine to investigate the cause for and the extent of the variation in results of anti-knock tests which arise when oil consumption is relatively high, a fact which has been observed by many users of knock testing engines. Arrangements were made to control and measure the air intake and oil sump temperatures. The tests were carried out at the air-fuel ratio required for maximum knock and at a knock-intensity which could be most suitably measured; the speed, intake air temperature, spark advance and water jacket temperature were kept constant throughout the series. Two lub. oils of 62 secs. viscosity at 210° F. were used, one prepared from Pennsylvania and the other from Coastal crude.

The gasolines used were (*a*) a str. run fuel of high anti-knock value; (*b*) a match for (*a*) in octane no. prepared from a str. run gasoline of low anti-knock value and tetraethyl lead; (*c*) as for (*b*) but using benzol instead of tetraethyl lead. The conditions for matching were a crankcase temperature of 110° F. and an oil consumption equal to ¼% of that of the gasoline.

The variations were assumed to be caused by (1) Partial admixture of lub. oil with incoming charge by leakage past the piston; (2) difference in cooling of piston due to different rates of heat transfer to cylinder wall. The effect of (1) was investigated by adding 4 and 8% of the 2 lub. oils to the gasolines and making tests at crankcase temperatures of 110° F. and 185° F., using a lub. oil consumption of ¼%.

Decreases in octane no. of from 1 to 3·5 at 110° F. and from 3 to 5 at 185° F. were found when 8% of lub. oil was added, the effect generally being slightly greater with Pennsylvanian oil. The effect of (2) was investigated by using lub. oil consumptions of ¼% and 2% and keeping the crankcase temperature at 110° F. The octane no. was reduced by approx. 0·7 at the higher consumption, and a wider variation may be obtained with fuels containing more benzol or tetraethyl lead.

To estimate truly the effect of the higher oil temperature, 2 secondary standard reference fuels and 2 other fuels containing more tetraethyl lead and more benzol than before, were rated directly in terms of pure iso-octane and normal heptane at 110° F. and 185° F., using a lub. oil consumption of ¼%.

The relative ratings were practically the same except for the benzol blend, which was 0·5 lower at 185° F. than at 110° F. The effect of oil consumption and temperature may be considerably greater if higher induction and cylinder cooling medium temperatures were used.

The conclusions are that (1) Errors due to different rates of oil consumption are approx. 1 octane no. in testing commercial fuels. (2) Errors due to variation in oil temperature may be as much as 1 octane no. (3) The mineral oil used does not affect conclusions (1) and (2).

The general use of a universal secondary standard would reduce differences in relative rating. It is recommended that all knock-testing engines should be designed and run at a minimum oil consumption and at as low and constant an oil temperature as is consistent with safety and good operation to improve accuracy, reduce carbon, and give steadier running. R. A. E.

See also Abstracts Nos. 502, 508, 562, 563.

Engines.

399. Temperature Distribution in Internally Heated Cylinders. A. B. Newman. *Ind. Eng. Chem.*, 1931, **23**, 29-32.

When a thick-walled hollow cylinder is heated all over its inner surface an immediate rise in temperature on the outer side is not observed. This lag is a function of wall thickness, rate of heat input, specific heat, density and thermal conductivity. The temperature at any radius will increase as time increases, and this paper offers a mathematical theory of heat flow in such cases. Curves and formulæ are given whereby one can ascertain the temperature at any radius at a given instant, if the above characteristics are available. Attention is also drawn to data available for application to problems of time-temperature relationships in bodies of various shapes which are heated or cooled externally. C. I. K.

400. Cold Starting Characteristics of Automobiles. A. J. Blackwood and N. H. Rickles. *J. Soc. Aut. Eng.*, 1931, **28**, 234-239.

A number of new cars have been examined as regards their starting properties in relation to the lubricant and fuel used. The cars were well run-in and then cleaned and adjusted to makers' recommendations before starting the experiments which were carried out in a special cold room, the temperature of which could be controlled from + 35 to + 120°F. A logarithmic chart is given which enables the viscosity at a given low temperature to be determined by extrapolation, when the Saybolt viscosities at, say, 100°F. and 200°F. are known.

The cranking speeds obtainable with oils of different viscosities at various temperatures were determined, and the starting characteristics of the car noted.

Results are given in graphical and tabular form for 17 cars (1930 and 1931 models) of varying number and capacity of cylinders. Minimum cranking speeds for starting were found to vary from 19 to 64 r.p.m., and maximum viscosities from 9,000 to 85,000 Saybolt sec. at the starting temperature. (The relation between cranking-speed and the log. of the viscosity for a given car and starting-motor is practically linear.) The cars range between the extremes of 9,000 and 55,000 sec. maximum permissible viscosity except in the case of one car of exceptionally good starting power, which would operate with oil of 85,000 sec. viscosity. (The cranking-speed of this automobile was then 40 r.p.m.) The present average normal limits for cranking speed are between 20 and 40 r.p.m., requiring that the oil have a maximum viscosity between 18,000 and 40,000 sec. at the starting temperatures.

The importance of the cranking-speed is shown by data relating to the number of attempts to start at certain speeds.

Assuming all cars would start if the oil viscosity does not exceed 18,000 sec., the lowest starting temperature is 25°F, while if 40,000 sec. is the maximum viscosity, 15°F. is the minimum permissible temperature, considering 14 winter grade oils marketed on the Atlantic coast.

The authors believe that the influence of distillation characters of gasolines is receiving undue importance, the important matters being design of carburettors, manifolds, and ignition, so that starting at low cranking speeds will be possible, and improving oils so that suitable viscosities at low temperatures can be obtained. Starting could not be obtained in some cases even although firing occurred. Car manufacturers should produce a starting-unit which will crank the engine at 35 r.p.m. at the lowest temperatures anticipated, and choke efficiency and ignition equipment might be improved.

E. B. E.

601. A French Diesel Engine. P. Legliso. *Aircraft Eng.*, 1931, **3**, 53-54.

The 100 h.p. Clerget heavy-oil engine is described. It is of the radial type and has the following characteristics:—Bore 120 mm., Stroke 130 mm., Weight 228 kg., Power 100 b.h.p. at 1800 r.p.m.

A special system of rollers by which the pilot unseats all exhaust valves simultaneously, allows any oil which may collect in the lower cylinders when the engine is at rest to escape. The decompression thus obtained is also useful in other ways.

The pistons are of light alloy while the cylinders and crankcase are of forged steel.

Turbulence is ensured by injecting the fuel to the centre of the hemispherical head of the piston, the eddies produced by the latter assisting good combustion, while no fuel comes in contact with the cylinder walls.

Each cylinder is provided with a fuel-pump which is controlled indirectly by a cam mounted on the crankshaft by helical splines which allows the timing to be varied. In addition each cylinder has an external device which allows the delivery to be varied relative to its pump. Means of making adjustments are indicated.

The atomiser consists of a small valve, in the head of which are shallow grooves almost tangential to the circumference of the exit.

Circulation of lubricating oil is by a double-geared pump, oil being delivered to the engine shaft by a special connection consisting of two cylindrical bushes fitting one inside the other.

As fuel, a light gas oil, S.G. 0·860, with a calorific value 9500 cal. is used, but the engine is adapted for any heavy fuel. The engine works on the four-stroke cycle and 150 h.p. has been obtained in tests. C. C.

602. Survey of Current Automobile and 'Bus Fuel-Line Temperatures. O. C. Bridgeman and H. S. White, *J. Soc. Aut. Eng.*, 1931, **28**, 315-320.

The frequency of troubles from vapour-locking in the fuel systems of automobiles has much increased in the last few years; accordingly data have been collected by the Bureau of Standards as to the temperatures attained at the various points in the fuel-lines in 'buses and automobiles.

For this work 27 cars and 8 'buses were used, temperatures being measured at such points as the fuel-tank outlet, vacuum tank inlet and outlet, fuel pump inlet and outlet, carburettor inlet and bowl, where these parts occur: 23 of the cars examined had fuel-pump systems, 3 had vacuum tanks and one a gravity feed. The cars had 4, 6, or 8 cylinders and all but one were 1929, 1930 and 1931 models.

The temperatures were measured by means of resistance thermometers, which were inserted into special pipe-fittings, the arrangements being described in detail.

Runs were made on each at speeds of 20, 40 and 60 m.p.h., until constant temperature was attained at the points of measurement. After the measurements were made the engine was allowed to idle until constant temperature was attained and the measurements were repeated. In some cases a run up a 6-mile hill was also included.

The results of the tests are given in graphs and tables, the following being the principal conclusions: The average temperature of gasoline in the fuel-tank is 18°F. above atmospheric. In going from the fuel-tank to fuel-pump or vacuum-tank the gasoline is heated another 11°F. Passing through the pump or vacuum-tank increases the temperature a further 12°F.; there is little change between the fuel-pump or vacuum tank and the carburettor.

The total rise is thus about 41°F. above atmospheric. Temperatures at fuel-pump and vacuum-tank are equally significant as regards vapour-lock,

while those at the carburettor are not very significant except possibly with V-type engines with the carburettor mounted between the rows of cylinders. On idling, a further 14 to 19°F. rise occurs, making about 60°F. in all. For the average car on the road in America it is concluded that to avoid vapour-lock at 40 m.p.h. the Reid Vapour-Pressure of the gasoline at 100°F. must not exceed 9.5 lbs. sq. in. About half the cars listed might be expected to give trouble under these conditions. In general, the rise of temperature is greater the greater the speed of the car. It is generally greater still after the hill-climb.

Very similar results are obtained for 'buses, but on the average the temperatures are somewhat lower than for cars, which is somewhat surprising considering the more limited space under the hood in buses.

One of the most important factors in design is the proximity of the fuel-line to the exhaust pipe.

It is considered that the introduction of the fuel-pump is more responsible for the increase in vapour-lock troubles than any increase in fuel volatility, and that the engine designer, by attention to points of detail, can reduce the trouble considerably. E. B. E.

General.

603. Progress in Prevention of Corrosion. Y. V. Kendall and F. N. Speller. *Oil and Gas J.*, 1931, **29** (41), 24.

A review is given of the more important recent developments of work on the cause, measurement and combating of corrosion, with particular reference to the problems met with in the oil industry. Corrosion is considered due mainly to the presence of potential differences whether induced by variations in concentration of oxygen or by contact between dissimilar metals. Anodic or cathodic polarisation and the presence or lack of protective films are also connected. Another factor is direct chemical action, as at high temperatures or by sulphur compounds in crude oil.

Of particular interest is the connection between the amount of film formation and the resistance to corrosion. A stable continuous oxide film on the surface of a metal prevents corrosion, while a discontinuous film causes pitting.

The following sources of corrosion have been investigated:—

(*a*) *Atmospheric Corrosion.*—The result of tests carried out by the A.S.T.M. on the corrosive resisting properties of commercial steels containing varying proportions of copper, phosphorus and manganese, showed that the formation of a copper oxide film on the metal greatly lengthens its life, while phosphorus, from about 0.08% upwards, improves the steel further still. Chromium, zinc and zinc alloy plating also increase the life of steels. It is stated that heating the chromium deposit for 30 mins. at 480° F. increases its resistance to corrosion sevenfold. Resistance to corrosion of plated automobile metals is measured in the laboratory by means of a calcium chloride spray or by refluxing with a constant boiling solution of pure nitric acid.

(*b*) *Underwater corrosion.*—In underwater corrosion, oxygen is the determining factor, the corrosivity being increased by pollution and by the chemical pretreatment usually given, which reduces the ability of the water to form protective coatings on metals. The addition of lime and soda helps the formation of these coatings by combination with soluble inorganic materials present. Portland cement is a good protector of metals against water corrosion. Before application to water lines the rust or carbonate scale must be

removed, *e.g.*, by solution in HCl (plus an inhibitor to prevent the metal being attacked). Steam and blowing with compressed air and abrasives is also used.

(*c*) *Boiler corrosion.*—The removal of oxygen by deaeration will eliminate most of the corrosion, with the exception of the "embrittlement" caused by mechanical stress and chemical action of sodium hydroxide in the boiler water. Deaeration is also sometimes assisted by the use of ferrous hydroxide to remove the last traces of oxygen. Certain methods of treatment of boiler water increases the tendency to produce embrittlement, while the presence of sodium sulphate will reduce it. Other inhibitors are phosphates, tannates, chromates, acetates, nitrates, etc.

(*d*) *Soil corrosion.*—The corrosivity of soils is tested by either chemical analysis or electrical tests. In the latter case the conductivity of the soil gives an indication of its corrosivity. No particular type of commercial steel is more resistant to corrosion by all types of soils than any other type, variations in the type of soil controlling the rate of corrosion. The use of different types of bituminous coatings is being examined.

(*e*) *Refinery corrosion.*—All classes of corrosion are met with in the oil industry, particularly atmospheric, soil, water and chemical action of H_2S. In drilling the muds used inhibit corrosion of the drill-pipes, etc., owing to the adsorption of finely divided material. The most serious damage to oil well equipment is due to the sulphur compounds in the salt water. A protective coating for tubing, rods, casing, etc., is therefore very desirable. In the absence of H_2S, the use of copper steels increases the life of the metal surfaces. In more severe cases galvanised tubing is better than steel or wrought iron, while copper-molybdenum iron and ingot iron are not superior. The addition of chromium and aluminium improves the resistance of the steel, while lining with portland cement is also an advantage.

In refinery work corrosion may be due to hydrochloric acid formed by decomposition of chlorides attacking the condensing equipment. Ammonia is used as a neutralising agent, special steels being also used with success for valves, fittings, etc. The corrosion by cooling water in condensers may be inhibited by use of sodium dichromate containing a smaller percentage of sodium phosphate or sodium silicate. Corrosion is also caused by the oxidation of metals on the firing side, and by the action of sulphur compounds in the crude oil. Neutralisation with lime, and the use of thicker and more resistant metals are employed to combat this. Reaction chambers are lined with portland cement concrete or other siliceous material.

Resistance to H_2S corrosion and direct oxidation is assisted by the use of steels containing 4% chromium, while nickel, used to stabilise the iron at high temperatures, sometimes reduces the resistance to corrosion.

Another important factor in refinery wasting of metal is erosion. This can often be reduced by the use of ferrules of special steel in the tube ends, or of free flow headers to reduce turbulent flow.

C. L. G.

604. Economics of the Crude Oil Potential in the United States. J. E. Pogue. *Petr. Div., Amer. Inst. Min. Met. Eng.*, 0.2. 1931.

The author concludes that the shut-in crude oil potential is the summation of the surplus initial production during the past three years, and has the effect of an inventory, being about one-half of that converted to actual production. The price of crude correlates with above-ground inventories corrected by adding the equivalent of the potential.

If all drilling ceased the potential would maintain production and demand in balance for 28 months. If all restrictions were removed, the U.S.A. production in 1931 would be 1.5 billion brls. and the price of Mid-Continent crude would decline to 10c./brl.

The potential is increasing, and must be reduced if higher crude oil prices are desired. Methods of control are thus at fault, and to succeed must involve a reduction in the economic effect of the combined inventory. G. S.

605. Stabilisation of the Petroleum Industry. L. Logan. *Petr. Div., Amer. Inst. Min. Met. Eng.*, 0.2. 1931.

Curtailment of crude oil production and gasoline production are pressing problems, and three major plans have been proposed: unit operation, proration, and an import tariff.

From an engineering and economic standpoint, unit operation is the soundest plan.

Proration is only a temporary measure, and as now applied does not rely on a scientific basis sufficient to make it a permanency. Many arguments have been advanced for and against the system.

The third plan is a temporary embargo on petroleum. The U.S.A. production is about 67 per cent. of the world's total, and its consumption about 63 per cent. of the world's demands. As long as it produces more than it consumes the U.S.A. is overproducing and, if an embargo is enforced, will lose most of its foreign markets. Therefore, production should be curtailed until production and consumption balance and temporary stability brought about.

The author suggests a Petroleum Dictatorship of three members to dictate when, where and how much oil is to be produced. One of their duties would be to enforce unit control of all fields, and to allow refineries to buy oil only from unit operated fields. G. S.

605. Compound Fuel. C. A. A. M. Roux. E.P. 315,006, 6.1.31. Appl., 6.7.29. Conv. (France), 7.7.28.

A synthetic fuel for gas producers is produced by intimate mixture of vegetable charcoal and mineral carbon, with a mucilaginous binder obtained from cellulose materials such as seaweed. An iron containing catalyst may also be added and gases incorporated by adsorption.

Pastes formed thus are distilled, the tar collected and used for further batches, and the distilled paste is quenched into an aqueous nitrate solution, thus furthering the reaction of the gas obtained in the producer. H. C. R.

607. Separating Device for the Separation of Oil from Mixtures of Oil and Water on Board Ships. A. Mejani. E.P. 340,549, 29.12.30. Appl., 27.8.29.

The object of the present invention is an apparatus for use on board ships for the removal of any kind of oil, from mixtures of oil and water, so that the exhausting pumps may discharge overboard water freed from oil, in order to prevent oil pollution in navigable water.

The improved apparatus is formed by a container, having an inlet duct leading to a baffle device for ensuring a steady inflow of the mixture into a settling chamber, an outlet duct for water near the top of the container, this outlet duct leading from a passage or chamber in communication with the bottom of the settling chamber, a rotating drum at the upper part of the separating chamber for collecting the oil from the surface of the liquid and a scraper for removing such oil and conducting it to an outlet duct leading to a recovery tank. H. E. T.

Coal and Shale.

608. Recent Literature bearing upon the Hydrogenation of Coal. D. G. Skinner. *Fuel*, 1931, **10**, 109–137.

Some of the chief difficulties in hydrogenation are the construction of high-pressure plant, the economic production of cheap hydrogen, the supply of heat, etc. All these points are covered.

The first part of the paper deals with catalytic hydrogenation and the effects of various catalysts on substances such as neutral oil from low temperature tar, primary tar, cellulose, Grozni mazout and acetylene, are included.

Further sections are on large-scale hydrogenation and general hydrogenation of coal. The properties of the oils produced are described in many cases together with methods of examination.

There is a section on hydrogenation of oil in which shale oil, petroleum asphalt, petroleum hydrocarbons and paraffin wax are discussed. In the first part of the article some 51 papers are abstracted. The second part deals with the patent literature, and 248 patents dealing with catalysts, plant and apparatus, methods of hydrogenation, indirect hydrogenation, combined cracking and hydrogenation, the use and reconditioning of hydrogenating gases and the pretreatment of materials used in hydrogenation process are abstracted. C. C.

609. Tar Removal by Electrostatic Precipitation. V. F. Gloag and J. P. V. Woollam. Fuel, 1931, **10**, 137—141.

While this article deals particularly with the removal of tar from coal gas or coke oven gas, it has also been successfully adapted for other problems, such as removing pyrites dust from sulphuric acid plant, acid mist from contact sulphuric plant, etc.

Electrostatic precipitation plants are simple and cheap to run compared with the usual impact, centrifugal or spray types of extractors, besides which they are far more efficient.

Essentially, the process consists in passing the gas through an intense electric field set up between a central high voltage electrode and the walls of earthed tubes or plates.

The H.T. direct current required is obtained from a step-up transformer supplying a suitable H.T. rectifier. The most satisfactory type of the latter —the Simon-Carves Static Copper Oxide rectifier—is described and illustrated. It consists of a series of copper discs coated with copper oxide and contained in two tanks filled with transformer oil, and depends on its action on the fact that the ratio of the resistance from copper to oxide coating is very high compared with the resistance from oxide coating to the copper.

Examples are given illustrating the efficiency and the economical working of this plant, the power consumption being less than 1 kw. per 100,000 cu. ft. of gas at N.T.P.

The precipitating chamber is of steel plates containing vertical 6-in. bore steel tubes. An electrode hangs down the centre of each tube. The gas descends outside and passes up the tubes where precipitation takes place, the particles coalescing on the walls of the tube. Suitable safety devices are installed. C. C.

See also Abstracts Nos. 506, 515, 577.

ABSTRACTS.

Geology.

610. Rusk-Gregg Field 60 to 90 Square Miles; Geological Features. W. C. Spooner. *Oil & Gas J.*, 1931, **29** (45), 30.

There are indications that the newly discovered East Texas Field (Rusk & Gregg Cos.) will comprise a producing area of 30 miles long and 2–3 miles wide, and this article briefly discusses some of its more important geological features.

The surface rocks belong to the Claiborne and Wilcox formations, and the sequence encountered in drilling extends into the Cretaceous from the Navarro formation to the Comanche Series. The producing horizons are the Austin Chalk and the Woodbine Series.

The structure is illustrated by two cross-sections through the field; east-west and north-south. The main features of the former are the sharp westward flexing of the Comanche surface and the diminishing dip shown in the Austin Chalk. The north-south section shows a marked flexure in the Comanche surface and in the Austin and younger Cretaceous; minor departures in strike and rate of dip are indicated, but no reversal of the latter.

Uplifts of these sediments in Northern Louisiana and Southern Arkansas followed the deposition of the Comanche Series, the Sabine uplift in N.W. Louisiana also coming into existence at this time as a distinct structural feature. The chief producing horizon on the Sabine uplift is a series of discontinuous sands at the base of the Tokio, previously referred to as Woodbine.

G. S. S.

611. East Hackberry Salt Dome, Cameron Parish, Louisiana. A. J. Bauernschmidt, junr. *Bull. Amer. Assoc. Pet. Geol*, 1931, **15**, 247–256.

This salt dome, which includes West Hackberry, was discovered in 1926 during a seismographic survey; surface indications are lacking. Although a true salt dome, it is not typical of those of the Gulf Coast, being much larger —approximately 6 miles long and 2 miles wide. East and West Hackberry are mere cupolas or knobs on this ridge-like salt mass, and these have been proved by seismographic surveys to be parts of the same mass separated by a saddle.

Most of the oil is obtained from Miocene Sands (about 3900 ft. thick), but the producing sands above the cap rock are probably Pliocene. The oil is typical heavy Gulf crude, ranging in gravity from 18° to 24° Bé. A list of 80 wells drilled at East Hackberry, together with maps and production tables, accompany the paper.

G. S. S.

612. Cretaceous Limestone as Petroleum Source Rock in North-western Venezuela. H. D. Hedberg. *Bull. Amer. Assoc. Pet. Geol*, 1931, **15**, 229–246.

This paper discusses the stratigraphy and lithologic characters of the La Luna and Cogollo Limestones, and of the origin of the oil occurring in them.

The La Luna Limestone is more thinly bedded and less resistant to weathering than the Cogollo, which is coarsely crystalline and in some instances almost entirely composed of *Exogyra*. Microscopic examination also shows that the two rocks are easily distinguished from each other; the former by

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its great abundance of small pelagic foraminifera (chiefly *Globigerina*), and the latter by its profusion of large fossils and entire lack of pelagic foraminiferal remains. Some of the Cogollo beds are composed mainly of calcareous algæ, such remains being characteristic of the formation generally.

Petroleum occurs in small quantity in both limestones. It is shown that the oil is indigenous in the La Luna (being diffused uniformly through the rock), whereas in the Cogollo it has migrated into the rock, occurring mainly in fractures.

Analyses also indicate that the La Luna Limestone is, or has been, the source of the petroleum, and the author concludes that its origin is connected either with the foraminifera themselves or with conditions particularly favourable to their existence and the subsequent preservation of their tests.

G. S. S.

Field Technology.

613. Wells Brought in Through Choke Nipples. K. C. Sclater. *Petr. Eng.*, 1931, **2** (6), 36.

When the Phillips Petroleum Company's Ciger No. 1 came in it produced more sand than any other well in the Oklahoma City Field. So great was the flow of sand that steps had to be taken to overcome the sand cutting of casing head connections and its attendant hazards.

Long choke nipples at the casing head and 12½ in. flow lines between the choke nipple and the traps were given a trial with very successful results.

The general type of vertical expansion chamber over the mouth of the well is dispensed with and in its place an extra large size flow line to the traps is used as an expansion chamber. Thus the chamber is horizontal instead of vertical. The oil and gas enter the flow line through a choke nipple. A 7 in. steel cross is screwed on the top of the well casing with one side outlet connected to the 12½ in. flow line by a flexible coupling. A hollow steel plug is screwed into the top outlet to take the impact of the sand-laden fluid in the casing. A 3 in. choke nipple about 2 ft. long is screwed into the side outlet of the steel cross. When in working position the choke nipple extends almost its entire length into the 12½ in. flow line.

To improve the control of the wells adjustable chokes have been used.

It was found that the 12½ in. casing became sand cut where the oil and gas emerge from the choke nipple on the down stream side, and this has been overcome by using about 2 ft. of 5½ in. drill pipe in place of the long choke nipple and inserting a short nipple, about 4 in. long in its place. This short nipple fits into a taper on the inside of the drill pipe on the upstream side. The choke can be quickly removed or replaced.

In addition to minimising sand cutting a decided improvement in gas oil ratios has been noticed.

L. V. W. C.

614. Experimental Measure of Slippage in Flow through Vertical Pipes. T. V. Moore and H. D. Wilde, junr. *Int. Pet. Tech.*, 1931, **3** (4). 15–23.

In order to estimate the amount of slippage in short gas lifts the authors have conducted tests and have shown that provided the fractions occupied by liquid and gas respectively are known, the slippage can be easily calculated. Unfortunately the results obtained did not apply accurately to actual wells, and the authors have concluded that flow in long pipes is more efficient than in the short pipes used for the experiments.

A number of equations are given to show the relationships of quantities and the final equation for the amount of slippage loss in foot pounds is :—

$$\frac{U_B}{U_G}\left\{ G\,P_{at}\,l_n\frac{P_B}{P_T} - K\,P_{at}\,L\,(P_B - P_T)\right\}$$

Where U_B = relative linear velocity, in ft./sec.
U_G = Linear velocity of gas, in ft./sec.
G = Rate of gas flow through pipe in cub. ft./min. at standard pressure.
P_{at} = Atm. pressure in lbs./sq. ins. absolute.
P_B = Pressure at bottom of pipe in lbs./sq. ins.
P_T = Pressure at top of pipe in lbs./sq. ins.
K = Henry's Law constant.
L = Rate of liquid flow in cu. ft./min.

The apparatus used was essentially a gas lift with a means for measuring the fraction of the pipe occupied by liquid under given conditions.

The liquid was contained in two measuring tanks 2 ft. 6 in. dia. and 5 ft. high. Connections were made so that liquid could be pumped alternately from the two tanks, and quick-closing valves were provided so that the change over from one tank to the other could be made instantaneously.

Two pumps were provided and the rate of liquid flow through the vertical pipe was regulated by means of a bypass line between the intake and discharge lines on the pumps.

Gas was supplied from the field fuel system and the volumes were measured by recording metric orifice meters. Five flow lines were provided : 4 in., 3 in., 2½ in., 2 in., and 1 in. Each line was composed of three joints of pipe making it 67 ft. 3 in. long. Near the bottom and top of each line, quick-closing valves were provided and by closing these valves simultaneously the oil in the pipe between them under any given set of conditions could be trapped and measured.

All pressures were measured by means of gauges which were calibrated at regular intervals.

During the experimental work five liquids were used, water, kerosine, heavy gas oil, light lubricating oil and heavy lubricating oil. Refined oil was used in preference to crude oil as weathering has little effect on the refined oil, but causes marked changes in the viscosity and gravity of most crudes.

The results obtained are tabulated. The authors conclude with the statement that while the results may probably be applied quantitatively to the design of short lifts, further work must be done in order to establish accurately the relationship between the various variables in active wells. L. V. W. C.

615. Direct Current in Oil Well Drilling. H. G. Dillon. *Oil and Gas J.*, 1931, **29** (46), 61.

The drilling of oil wells by electric power, where the energy is generated at the well by means of suitable generators connected to internal combustion engines, has many advantages. The efficiency of the engine, the small amount of water required, the flexibility of the electric drive and the adaptability to this work are the main factors with this new drive. The high speed light weight Diesel Engine has provided an excellent prime mover for this service.

Mechnical equipment, consisting of clutches, gears, chains, etc., give an overall full load efficiency of 94 to 98% but the highest overall efficiency obtainable with a combination generator and motor hookup will not exceed 85%. It would appear, from these figures, that the engineer would be justified in classifying the Electric Drive as unsuitable, but full load efficiency figures are very misleading.

If the load was continuous and of constant direction, h.p., and speed and concentrated at one point it is probable that the best method of power transmission would be the mechanical connection. If the load is reversing, is not continuous, and consists of various values of torque and speed, electrical transmission has advantages.

Where an engine is connected to a load by mechanical means the ratio of the speed of the driven shaft to the engine speed is fixed in the gearing used. Changes of gears can be made, but the average drilling equipment used has so little inertia that it is almost impossible to shift gears while engaged in the hoisting cycle.

A graph is given showing a true comparison of electrical and mechanical transmission, where the speed of the driven shaft is plotted in terms of % of full load engine speed against the torque exerted in the driven shaft in terms of % of full load engine torque. The curves represent the hoisting cycle that might be obtained with an infinite number of shifting gears for each efficiency as indicated from a 375 h.p. engine. Curves are plotted for 100, 95 and 85 % of transmission.

The motor speed characteristic shown is one that may be obtained from a properly designed D.C. equipment being driven by the 375 h.p. engine.

The shortcomings of the mechanical connections are best shown by considering the equipment to be operating on a hoisting cycle where the starting torque will be 200% of full load torque with the normal running torque being 100% of full load torque. With the mechanical transmission a set of gears must be used where the speed of the driven shaft would be limited to approximately 50% of the normal full load speed and the torque exerted increased 200% normal full load torque, always assuming that the clutch will have sufficient capacity to accelerate the load at this high value of torque without stalling the engine. After the equipment has been accelerated to normal running speed, the speed of the driven shaft in terms of % full load engine speed will be about 49% with 100% of torque exerted in terms of % of full load torque. Thus, no matter how the running torque may drop, the speed of hoisting is fixed by the gears used in starting and accelerating the load. The full load efficiency of the drive may still be 95%, but the engine will be operating at approximately one half load and the load will be moving at approximately one-half of the speed that it would move if the additional engine horse-power could be applied to it.

With the D.C. motor drive the 200% full load torque may be exerted at the start and as the load falls off the speed of the motor will increase so that with the conditions assumed the driven shaft will rotate at approximately 85% of normal full load speed. At this point the engine would be operating at full load and the load would be travelling at twice the rate of speed that could be obtained by mechanical transmission.

It will be seen, then, that a properly designed D.C. circuit will perform the function of a clutch and of an infinite number of shifting gears with fine speed control at all torques and speeds permitting the engine to operate at a high load factor at all times.

With the A.C. system these advantages do not exactly apply. The A.C. system is a constant voltage system and the torque exerted by the motor therefore governs the current flowing in the circuit. Thus the engine output is governed by the motor output. The A.C. equipment then takes the place of the clutch in the mechanical hookup and provides fine speed adjustment at any value of torque within the capacity of the engine without varying the engine speed.

With the electrical equipment the clutch is eliminated and the engines may be installed in any position either inside or outside the rig.

The decision as to whether D.C. or A.C. should be used should only be made after a careful study. A.C. has the distinct advantage of high load efficiency and low line losses when it is desired to transmit power any great distance. This enables a central power station to operate rigs situated at any distance.

The full load efficiency of D.C. is lower than that of A.C., but if any A.C. equipment is operating at a point of the controller other than the highest point the efficiency is less. Higher efficiency may be obtained from direct current drive for all speeds less than full load speeds.

In general for drilling equipment where the power is supplied by internal combustion engines, direct current equipment represents the best method of power transmission between the engine and the driven load. L. V. W. C.

616. Unique Stations Double Line Capacity. W. F. Lowe. *Int. Petr. Tech*, 1931, **8** (4), 46.

The electric pump station capable of handling 75,000 brls. or more per day is a recent addition to the pumping industry and one of the outstanding examples is the station recently put into operation by the Texas Empire Pipe Line Co.

This station is equipped with 3 motor driven centrifugal pumps, any two of which, when hooked in series, have a maximum capacity of 94,000 brls. per day at 750 lbs. pressure. This represents the maximum specification, but at the present time the station is working to a capacity of 73,000 brls. per day.

The centrifugal four-stage pumps are special oil line type, with expanding loop design, having a 10-in. suction and an 8-in. discharge. Each is equipped with an oil-cooled thrust bearing and has one stuffing box on the suction side. The pumps weigh approximately 20,000 lbs. each and are 8 ft. high by 5 ft. wide.

They are directly connected through flexible couplings to 800 h.p. three-phase, 60 cycle, 2200 volt synchronous motors having a unity power factor and a temperature rise above the surrounding temperature of 40°C. Collector rings are entirely enclosed and gas proof, eliminating the necessity of a fire wall between motors and pumps. The speed of the motors is 1800 r.p.m. Three control panels are provided, one for the incoming power line, one for the exciter, and the third for the three motors.

A special set up drum switch provides for three different series hookups with the motors and a signal light mounted miniature piping layout of the station indicates by a red and green light the position of the valve and pumping units which are in use. This feature is one that will be used in the semi-automatic remote control of some of the equipment. Though the entire station will eventually have remote supervision only the major units will be directly controlled by an operator at a distant panel. These units will be the exciters and motors and the motor operated valves, and all may be started and stopped or opened and closed by electric impulses sent by wire. The operator will have supervision over suction and discharge pressures, low voltage, etc., but will not have control.

A mishap, such as a hot bearing, a blown gas gasket or any difficulty concerning those points over which he has supervision, will be indicated on the control board. The various units are so connected through a system of relays and circuit breakers that any difficulty reflects itself immediately in stopping the motors.

The sequence of operation at present is simple since the station is but semi-automatic. The suction lines to be used are opened first, and the oil is by-passed through a small line around the discharge gates as a pressure equaliser to facilitate their being opened. The opening of these valves is manual, but the remainder of the sequence is from the control board.

The motor operated suction valve is opened by depressing a push button, since this valve is interlocked with the remainder of the control. As soon as this valve reaches the open position, which is indicated by the flashing of a lamp, the direct current exciter set may be started. This permits the operation of the main motor control board and a second rotary selector switch is then set for the motors.

Depressing another push button starts one motor on reduced voltage. Connections automatically changed bring on full voltage and finally apply the excitation and synchronise the motor on the line. Immediately following this operation the sequence repeats itself for the second motor without further attention from the engineer. L. V. W. C.

617. Standard Operating Practices Employed in Van Field. W. L Baker. *Petr. Eng.*, 1931, **2** (6), 30.

In the Van Field, East Texas, all operating practices are standardised; all wells are drilled, completed, produced and equipped alike.

Five companies own an interest in this field and the attention that has been paid to the various operating practices has resulted in one of the most immaculate oil properties in existence.

When the wells are finished the derricks are removed and all connections are housed in a neat structure. A control arm, operated from outside, obviates the necessity to enter the housing to open or close the well.

The rotary system is used throughout this field and the average producing depth is 2500 ft. The bottom hole pressures are about 1250 lb., with a tubing pressure of 400 lbs. The rotary mud is maintained at 10 lbs. for drilling and 9½ lbs. per gal. for drilling in.

The steel derricks are 96 ft. high, and for producing the reinforcements are removed.

The surface pipe is 10¾ in. and is set and cemented at 300 ft. The 7 in. string is set in the top of the Woodbine horizon and cemented, the cement being allowed to stand 4 days. Coring operations are started in the Eagleford above the Woodbine in order to definitely determine the depth of the Woodbine. As soon as the Woodbine is reached the final string is set.

Holes are not allowed to vary over 5 ft. from the vertical and instruments are run every 40 ft. to determine deviation. If the hole is found to be more than 5 ft. from the vertical it must be straightened before additional drilling is done.

The producing horizon is drilled to a predetermined depth with rotary tools, after which all wells are completed through 2½ in. tubing. A ⅛ in. liner sufficiently long to extend into the casing is set on bottom, and after setting the hole is washed clean with water. Most of the wells come into production without swabbing after being washed.

All wells are equipped with 3 in. Christmas trees, 6½ in., 2500 lb. master gates and a tubing head gate that permits retraction of tubing. This gate is operated by remote manual control. To permit the cleaning out of the wells through either the casing or the tubing a 2½ in. gate on a short length of 6 in. pipe is secured in the 10 in. casing just above the ground.

The flow line gate valves are of 3 in., 2500 lb. construction. A positive bean type choke on the flow line on one side of the head and a regulating choke on the other side are provided on the hookup. The positive choke is used for taking all potential gauges and the regulating choke for controlling the prorated flow of the wells.

Daily readings are taken of tubing and casing pressures and of oil temperatures. The correct temperature readings are secured by means of an installation that does not permit the gas to expand and consequently lower the

temperature. A small pipe is welded into a nipple that extends into the flow line and a small hole, large enough to hold a thermometer, is drilled into the projecting plugged end of this pipe.

Individual separators, set on pedestals at sufficient height to permit atmospheric pressures and still dump the oil into tanks by gravity, are used at all wells. L. V. W. C.

Crude Oil.

618. Vacuum and Chemical Process for the Dehydration of Emulsions. *Petr. Eng.*, 1931, **2** (6), 182.

The process used by the Lion Oil Refining Co. at El Dorado (Ark.) for dehydrating Smackover crude, which contains 4 to 8% of B.S. and W., involves the heating of chemically treated oil in a pipe still to temperatures of 220–230° F. from whence it is passed to a dehydrator tower maintained under a vacuum of 75–150 mm. abs. pressure. The throughput of the dehydrator plant is rated at 6000 bls./day. No trouble is experienced from salt deposition in the tube owing to the low operating temperature of the furnace. When the dehydrated crude is charged direct to a distillation unit no economy can be expected from the point of view of utilising the heat available in the dehydrated crude owing to its low temperature, as compared with that of an atmospheric dehydration plant. Savings are, however, effected owing to the sensible heat required to raise the water in the crude to the higher temperature. When operating as an independent unit the vacuum dehydrator shows considerable saving in fuel over the atmospheric dehydration plant. Benefit is also derived by preventing the deposition of B.S. in tanks, pipe lines, etc.

A diagrammatic sketch of the vacuum dehydrator unit is given.

C. L. G.

619. Vaporisation of Petroleum and Pressure Distillate. G. H. Fandrer. *Petr. Eng.*, 1931, **2** (6), 176.

An investigation has been made with a view to obtaining information on the results of equilibrium single-flash distillation, since this practically represents operating conditions in a pipe still.

The two limiting types of vaporisation are (1) equilibrium flash vaporisation, in which all the vapour and residue formed are maintained in intimate contact until separated under chosen equilibrium conditions, and (2) differential vaporisation in which vapour is formed and removed in successive infinitesimal amounts, the summation of the differential increments giving the amount of vapour required. All vaporisation processes work between these limiting methods. Information regarding the differential vaporisation of a substance can be obtained from a consideration of its true b. pt. curve.

The four following materials were flash vaporised under equilibrium conditions at various temperatures and the properties of the resulting distillates and residues determined: (1) mixed base Mid-Continent crude, (2) cyclic-base Californian crude (Inglewood field), (3) pressure distillate from shell pressure still, and (4) pressure distillate from a pipe still cracking plant running a heavy fuel.

The apparatus used was that described by Leslie and Good (*Ind. and Eng. Chem.*, 1927, **19**, 453).

Full details are given in graphs and tables of the true b. pt. curves of single-flash products at different temperatures from the four types of material examined (all data being corrected for distillation loss by the usual displacement of the curve). Figures showing the yields from the crude, and properties of the crude, distillates and residues are also given. C. L. G.

620. Distillation of Mineral Oils by Highly Super-Heated Steam. C. Ab-der-Halden. *Rev. Petrolifère*, 1931 (416), 365–370.

For the distillation of crude mineral oils the temperature of distillation is kept as low as possible by reducing the pressure or introducing steam. It is possible to obtain satisfactory results by admitting steam and oil at the same time into a heated retort, but on a large scale technical details make this impracticable.

In the plant described, preliminary heating is by heat exchangers and then by a tubular system, the hot oil and super-heated steam being introduced into a still maintained at the required temperature. This temperature is important as the composition of the residue and the vapours depend on it.

The theory of steam distillation is briefly discussed, and the analogy between fractional condensation and evaporation is indicated.

A diagrammatic sketch of the plant is given. C. C.

621. Distillation. R. D. Hunneman, F. M. Rogers and R. E. Wilson, Assrs. to Standard Oil Co. U.S.P. 1,791,209, 3.2.31. Appl. 1.4.25.

A method of distilling heavy hydrocarbon oils is described in which the oil is brought to a temperature of 670° to 760° F. and mixed intimately with steam in the proportion of 2 to 10 lb./gal. of distillate produced. The oil is passed through an unheated conduit at a velocity sufficient to prevent separation so as to ensure thermal equilibrium in the mixture. The latter is discharged from the passage into the vapour space of an enlarged chamber, where the vapours are separated from the vaporised oil. The vapours are retained in the chamber for not more than one second, whilst an absolute pressure of 25 to 75 mm. is maintained in the conduit and in the chamber. W. S. E. C.

See also Abstract No. 623.

Natural Gas, etc.

622. Removal of Dust from Natural Gas Pipelines. W. J. Miskella. *Oil and Gas J.*, 1931. **29** (44), 56.

Dust entrained from the wells and from the rust occurring in pipelines causes damage to plant and increases transport charges in the long distance transmission of natural gas. A separator for removing this dust is described. The gas encounters a stationary perforated plate, giving it a whirling motion. The hub of the plate acts as a vortex breaker, the dust is thrown outwards, and its progress retarded by a rib further down the separator. The dust is then directed into a dust pocket made of steel pipe with a welded head, and fitted with a discharge pipe. An ejector arrangement is provided by which the dust is lifted when the gate valve on the discharge pipe is opened. The dust can be caught in a bucket with practically no loss of gas if the gate valve is closed again as soon as the gas commences to blow through.

Diagrams of the separator and of the pipe connection are provided.

R. A. E.

623. Characteristics of Hydrogen Sulphide. C. K. Francis. *Oil and Gas J.*, 1931, **29** (42), 153.

The chief physical characteristics of hydrogen sulphide are: Sp. Gr. (air=1), 1·1906, (water=1), 0·9; B.P., −76° F.; M.P., −117° F.; Sp. Heat (const. press.), 0·2423; critical temp., 212·7° F.; critical pressure, 92 atm.; solubility in water, 4 vols./1 vol. water. Hydrogen sulphide reacts explosively with excess oxygen to give SO_2 and H_2O, but with insufficient oxygen, free sulphur is formed.

The H_2S content of natural gas varies from 0·6 to 6%, while that of refinery gases when working up Mexican crude may be up to 12% by volume. It may be detected by its action on paper moistened with lead acetate and on copper strip, and may be estimated by titration with iodine. (Full details of the method of carrying out these tests are given.)

The toxic nature of H_2S requires the taking of great precautions, a concentration of 0·005% being sufficient to cause irritation and inflammation of the eyes, throat, lungs, etc. Exposure for 5 mins. to an atmosphere contaminated with 0·05% H_2S may prove fatal. Fresh air and artificial respiration should be given immediately signs of H_2S poisoning are apparent.

H_2S is also dangerous when taken internally, owing to the formation of sodium sulphide, which is caustic towards flesh. The sodium sulphide on entering the blood stream decomposes, liberating H_2S, which reacts with the iron in the blood.

Hydrogen sulphide is a frequent cause of fires in refinery equipment owing to the pyrophoric nature of the iron sulphide, readily formed by its action on iron.

In the presence of oxygen, H_2S tends to form sulphuric acid and in the absence of oxygen liberates free sulphur, particularly at high temperatures, with detrimental effect to refinery equipment. The removal of H_2S from natural gas transported in pipelines considerably lengthens the life of such pipes.

The removal of excessive quantities of sulphur compounds in crude can be effected by hydrogenation, particularly in the presence of a catalyst. Various methods of removing H_2S are suggested :—

(*a*) Washing with water, caustic soda solution, iron oxide and hydrate, sodium carbonate (N.B., details of the Koppers method using this reagent are given), hydrated lime and milk of lime.

(*b*) Absorption with triethanolamine, from which it can be removed by distilling at 100° C.

A suggestion is made (Bur. Standards Circular 2847) that crude oils containing H_2S could be purified by treatment with gaseous SO_2, yielding free sulphur and water. The sulphur would have a marketable value, while the oil would be more valuable on account of non-corrosivity. The same process could be applied to hydrocarbon gases containing H_2S. No tests have yet been carried out on oils containing large quantities of H_2S to verify this suggestion. C. L. G.

624. Extent of Butane Extraction Depends Upon Many Factors. R. A. Boyd. *Oil and Gas J.*, 1931, **29** (45), 122.

Since the advent of fractional analysis it has been found that a natural gasoline having a 90% distillation recovery and E.P. of less than 305° F. can contain 40/50% of butane. The exact percentage depends on the ratio of iso- and n.-butane, and on the ratio of pentane and hydrocarbons of higher B.P. present, when considering propane-free gasolines. As the E.P. increases the allowable percentage of butane for a given recovery diminishes, but an E.P. in excess of 290/320° F. indicates topping of absorption oil or addition of other high E.P. products. The old specifications for natural gasoline, by including maximum A.P.I. gravity limits, caused a loss in production and a high recovery on distillation of the gasoline. From a knowledge of the composition of the gas and of the amount of butane which may be added to the hydrocarbons of higher B.P. present in order to yield a gasoline of 90% recovery, the percentage of the butane which should be extracted can be determined. Obviously it is uneconomical to extract more butane than can be utilised, but the extent of extraction can be controlled by plant design and rate of oil circulation.

Therefore, in order to determine the oil/gas ratio for plant operation it is necessary to ascertain: (1) the percentage of butane that can be retained in the gasoline; (2) the analysis of the gas; (3) absorber pressure; (4) Mol. wt. and absorption factor of the absorption oil; (5) temperature of oil leaving the absorber; (6) the vapour pressure of n. -butane at the temperature of the oil leaving the absorber. R. A. E.

625. Working Up Spent Gas-purification Material to Pure Tar-free Sulphur and Tar-free Purification Material Capable of Being Used Again. Ruhrgas A.-G. E.P. 342,611, 5.2.31. Appl., 29.4.30.

The spent gas-purification material is first treated with benzol or heavy benzol or toluol in the cold, thus removing the tar. The sulphur is extracted with the same solvents—*i.e.*, benzol, toluol, carbon disulphide, tetraline, etc.—at raised temperatures, especially above the boiling point of the extracting medium under pressure. W. S. E. C.

626. Decomposition Under the Action of Heat of a Mixture of Methane or Other Hydrocarbons and Water Vapour. Union Chimique Belge, S.A. E.P. 343,172, 3.2.31. Appl., 3.9.29.

This invention relates to the endothermic decomposition under the action of heat and without the use of a catalyst, of a mixture of water vapour, methane or gaseous or volatile hydrocarbons such as those contained in natural gas or in gases obtained from the carbonisation of fuels. The gases to be burnt to supply heat for the reaction are admitted direct without preliminary heating into a reaction chamber or conduit. The air for combustion is heated in one regenerator for one period, and the burning gases escape from the reaction chamber or conduit through the other regenerator, the treated gases traversing during the next period both regenerators and the reaction chamber or conduit in the opposite direction. W. S. E. C.

Motor Fuel.

627. Correlate Vapour Pressure and Losses. W. K. Lewis and S. P. Coleman. *Oil & Gas J.*, 1931, **29** (42), 130.

Some samples of vapour displaced by incoming crude in the tanks of a pumping station have been examined to ascertain the total percentage of hydrocarbon vapours and of each individual hydrocarbon present. These results are tabulated, and compared with the percentage estimated from the known vapour pressures of the crudes applying Dalton's law. The calculated percentages agree closely with the determined figures. From the known throughput of oil and the data obtained it was demonstrated that measures for prevention of loss were amply justified.

Applying this information to a consideration of the condition obtaining in gasoline storage tanks during storage, pumping and filling and to the effects of change of atmospheric temperature it is deduced that windage effect and loss by "breathing" of tanks are proportional to the vapour pressure of the gasoline, other things being equal. Minor losses, such as those experienced in filling tanks and tank cars under various conditions are calculated to be proportional to the vapour pressure or to a higher function of the V.P. and also to the mol. wt. of the gasoline vapour. To test the validity of these deductions a large scale test was made on five gasolines of vapour pressures varying from 204/498 mm. Hg. at 76° F., quantities of 10,000 brls. being used for each test. The total losses due to refinery storage, loading, shipment by water, unloading, bulk sales storage and local distribution were measured,

samples were taken at various points and tested for vapour pressures, propane and butane content, distillation range, etc. The losses plotted against the average vapour pressures during distribution show that up to 300 mm. the loss is substantially a linear function of the vapour pressure at the existing temperature. For gasoline of higher vapour pressure the loss is proportionately greater.

Experiments have been made which show that the effect on vapour pressure of the addition of known amounts of butane to a gasoline can be calculated by Raoult's law within an accuracy of 3%. Making due allowance for the differences in distribution conditions it is thus possible for a refiner, having a knowledge of the losses experienced in his deliveries and of the vapour pressure and the propane and butane content of his gasoline, to estimate the increase in loss which will be experienced if the vapour pressure of his gasoline deliveries is increased. It is also possible to estimate whether the installation of recovery equipment is economically justifiable. R. A. E.

628. Value of Colour and Gum Inhibitors. H. W. Sullivan. *Oil & Gas J.*, 1931, **29** (**42**), 156.

Excessive gum content is undesirable in a gasoline, but removal of colour and gum from a cracked spirit by treatment is expensive and causes a deterioration in anti-knock value. The action of inhibitors on gasolines prepared from a Dubbs pressure distillate have been examined. The samples represented: (1) Untreated gasoline from untreated pressure distillate, (2), (3), (4) and (5) equal cuts from (6), which was a 425° E.P. gasoline from treated pressure distillate, (7) sample (6) treated with acid and clay. Anthracene, gum camphor, an inhibitor known as Du Pont No. 2 and aniline were added to separate portions of these gasolines in the proportion of 1 lb. to 8,000 gal. and deterioration of colour on standing in sunlight, dissolved gum by Selminite dish method, and Ethyl Gasoline Corporation accelerated ageing tests determined on the whole series. The effect on colour stability varies according to the gasoline, but anthracene and Du Pont No. 2 generally cause an improvement. Aniline accelerates colour formation so that gum and ageing tests were not carried out on the aniline series of samples. All the other inhibitors decrease the gum content of the untreated sample, but whereas with the treated samples Anthracene and Du Pont No. 2 generally give improvement, gum camphor accelerates gum formation. The accelerated ageing tests revealed wide variations in the effect of the inhibitors, but on the other hand an experiment carried out on a sampled store for ten months and which, according to the ageing test, should have shown excessive gum after this period, indicates that the method does not provide results which can be relied upon to predict the effect of actual storage on the gum content.

Pressure distillate is generally treated with acid of 50/93% strength, followed by water and caustic washes.

It is generally agreed that acid acts on unsaturated compounds forming (1) neutral or di-alkyl esters which are oil-soluble and then (2) acids of mono-alkyl esters, which are water soluble. The neutral esters decompose on redistillation and corrode the vapour lines and condensers so it was thought that colour might be related to acid formation on distillation. The pressure distillate was therefore treated with 93% acid in quantities of 1/40 lb. per brl., and one test made with acid of 98% strength. The temperature at which acidity developed on heating, together with the colour and gum content of the resulting gasolines were determined. The results show that (1) gasoline properties and neutral ester formation depend to a greater extent on acid strength than on the proportion used (2) for maximum colour of the gasoline there is an optimum percentage of acid (3) gum is eliminated by large quantities of acid. R. A. E.

629. Purification of Pressure Distillates. E. H. Potthof. *Oil & Gas J.*, 1931, **29** (42), 141.

An investigation has been made of the effect on the properties of 405° endpoint gasoline ex Dubbs pressure distillate, of refining with different quantities of both 60° Be and 66° Be sulphuric acid. The effect of varying the time of contact and the sludge and polymerisation losses have also been studied. Tables showing the properties of the gasoline refined under the different conditions are given.

The gasoline is acid treated at 60°–70° F., settled, neutralised with 10% of 10° Be caustic soda solution and redistilled. The sludge loss is taken as the volumetric loss of the distillate, corrected for evaporation during treatment, but does not include neutralisation losses, owing to the difficulty of measurement. For this reason the losses given will be somewhat smaller than those found in actual practice. The polymerisation loss is taken as the residue after distillation of the treated gasoline less the residue from a blank distillation of untreated gasoline. The quantity of "polymerised hydrocarbons" is undoubtedly lowered by solution in the acid sludge but increased by the sulphuric acid combination products soluble in oil. Reference to the tables shows that the use of 10 lb. 66° Be acid per brl. reduces the benzol equivalent from 47 to 43, while 20 lb. acid lowered this value to 33. The sludge and polymerisation losses also reach a considerable magnitude with acid treats of over 5 lb./brl. Increasing quantities of acid have a greater effect on the lower boiling hydrocarbons than on the higher boiling, while the gum contents (copper dish) are greatly reduced.

Similar tests using 60° Be acid showed that the resulting loss is about three-quarters of that when using 66° Be acid. When using 5 lb. acid the chief advantage to be gained by using 66° Be acid is in the reduced losses, but with larger quantities of acid there is a decided reduction in the loss of anti-knock value.

Increasing the time of contact with acid has more effect on the loss than on the quality of the final product. With 66° acid, increasing the time of contact from 2 to 30 min. almost triples the sludge and polymerisation losses, while with 60° acid the sludge loss is only doubled and the polymerisation loss slightly increased. Very little alteration to the properties of the final product is caused but if more than 5 lb. of acid is used the effect of increasing the time of contact would be much greater. C. L. G.

630. Knock Rating and Distillation Range. D. P. Weld. *Oil & Gas J.*, 1931, **29** (42), 98.

The possibility of eliminating certain cuts from gasolines in order to improve the anti-knock quality of the final product has been investigated. The gasoline examined was a product obtained by liquid phase cracking of a Mid-Continent gas oil. Since the separation of a number of narrow cuts would present difficulty in practice, the gasoline was distilled into seven fractions, the distillation ranges and gravities of which are given. The fourth, or middle fraction, comprised 47% of the original gasoline. To eliminate the effect of distillation loss a blend of the fractions in the percentages obtained was used as a standard of comparison. Blends of various consecutive fractions were then made, and examined for anti-knock value, distillation range and gravity. The results are presented in both tabular and graphical forms. The octane numbers vary from 50 to 61, the standard having an octane number of 55, so that an increase in octane number of six could be obtained by sacrificing 24·9% of the heavy ends of the gasoline. There was no indication that any of the fractions had any unexpectedly high knock-promoting or knock-suppressing properties. R. A. E.

631. Final Report of the Departmental Committee on Ethyl Gasoline. A. G. Francis, Dixon, R.A. Hopple. Ministry of Health, Gt. Britain, 1930. 91 pp.

It is concluded that no public health reasons exist for prohibiting the use of ethyl gasoline in automobiles so long as reasonable precautions are taken. The recommended precautions include labelling the cans and pumps, and dyeing the gasoline, which should not contain more than 1 pt. of PbEt in 1300 pts. by volume or about 1 in 650 by wt. A new and accurate method of detecting the presence of small amounts of lead is described. By use of the method, the lead in urine, dusts and in garages was determined. More lead was found in crank-case oils in automobiles using ethyl gasoline. No amount likely to create danger was found in lubricating oils, either as a result of spilling or from the exhaust gases in city traffic. By animal experiments, the daily application of ethyl gasoline to the skin caused neither local irritation nor impairment of the general health. Only under the most exceptional circumstances is there any danger from ethyl gasoline exhaust gases in garages or on city streets. W. S. E. C.

632. Refining of Motor Benzol and Other Fuels. The Instil Process. *Gas World*, 1931, **94** (2431), 12–16.

The Instil process aims at the elimination of the use of sulphuric acid in the purification of crude benzol, low-temperature oil, shale-oil, etc., intended for use as motor fuels. This process increases yields of finished products by the removal of only a part of the unsaturated hydrocarbons; *i.e.*, the gum-forming diolefines which are easily polymerised. (Acid refining removes also the useful olefine content, diminishing the anti-knock value of benzol and the easy starting properties.) The amount of carbon-disulphide and thiophene is not appreciably reduced, but a modification has been developed which will remove the former at minimum cost and trouble. Existing refining plants can be adapted with little cost to the Instil process, by the addition of a filter, neutraliser and heating coil to the washer.

Crude Benzol, the spirit stripped from gas from high-temperature cooling plants, town gas plants, etc., is made up of (1) motor spirit up to 140° C.; (2) light solvent naphtha 140–160° C.; (3) heavy solvent naphtha 160–190° C.; (4) bases and tar acids; (5) residues consisting of naphthalene and stripping oil; (6) gum-forming and objectionable sulphur compounds.

Low-Temperature Oil.—Crude spirit stripped from gas from low-temperature coal distillation, plus first runnings in the distillation of the tar. It comprises (1) motor spirit up to 170° C.; (2) bases and tar acids; (3) residues; *i.e.*, stripping oil; (4) impurities as for benzol.

Shale-Oil.—Crude spirit from shale—similar to low-temperature oil.

Miscellaneous Crude Spirits.—Comprises cracked spirits and crude petrols, etc., consisting mainly of motor spirit and impurities.

This article deals mainly with the refining of coke-oven benzols. Briefly the process is as follows: The crude benzol from the de-benzolising still once run if necessary, is freed from pyridine and like bases by treatment with sulphuric acid or in a continuous pyridine scrubber on its passage to the washer. (The bases do not usually exceed 0·5% of the crude benzol.)

The base-free spirit is treated at 55° C. with 2–3% of Instil refining medium. After agitation at this temperature for 90 min., the benzol and entrained medium is filtered and neutralised, passing to the still for rectification.

Benzol refined by this process will pass N.B.A. Specification, excepting the acid test, which is simply a confirmation of acid washing. In place of acid test two others are suggested: (1) The aluminium chloride test, and (2) oxidation test.

The practical advantages obtained from the process are: (1) increased yields, (2) freedom from free sulphur, (3) practically gum free, etc.

Data is given of engine tests, which indicate that acid refined benzols produce 21% more carbon in the engine than Instil refined. Also a comparison between a low-temperature spirit Instil refined and an ordinary No. 1 Petrol shows that anti-knock and power-increasing values of the unsaturated hydrocarbons in the former remain unaltered.

The Instil refining medium consists of: (1) Ferric sulphate, which polymerises the diolefines and has a catalytic and oxidising effect; (2) Absorbent earth which keeps the medium in fine state of division also absorbs sulphur; (3) Free sulphuric acid which amounts to 4–5% of medium and is present to prevent the ferric sulphate becoming basic on storage.

The spent medium is a dark-coloured, inoffensive powder easily disposed of, a more desirable waste product than the noxious acid-tar obtained with acid refining. W. A. W.

633. Treatment of Petrol and other Organic Liquids Containing Water. R. Lant and W. Koreska. E.P. 343,105, 10.2.31. Appl., 10.9.29.

Water is removed from petrol or organic liquids by filtration through cellulose esters of the higher fatty acids. The filter may be built up in any of the standard forms. T. A. S.

634. New Fuel for Internal-Combustion Engines. H. Menz. E.P. 343,238, 19.2.31. Appl., 14.12.29.

The fuel consists of alcohol 50 pts., benzol 10 pts., tetrahydronaphthalene 5 pts., distilled water 30 pts., pulverised sulphite cellulose extract 1 pt., sulphonated whale oil 4 pts. The material gives a stable emulsion and does not cause rust. T. A. S.

635. Producing Gasoline. D. G. Brandt, assr. Doherty Research Co. U.S.P. 1,791,113, 3.2.31. Appl., 25.2.26.

Petroleum vapours from the cracking and distillation of petroleum are passed through a series of three condensing zones in which they are maintained under a pressure of about 50 lb./sq. in. The first zone is maintained at about 250° F. and the second at about 150° F. By this means only relatively high boiling point constituents are condensed in these zones. The temperature in the last zone is maintained at normal atmospheric temperature. The condensate from each zone is cooled separately and all three are then mixed and stored under pressure. W. I.

See also Abstract No. 670.

Kerosine.

636. Lamps for Heating. Shell-Mex Ltd. and A. W. Richardson. E.P. 342,748, 5.2.31. Appl., 5.10.29.

This invention relates to an improved lamp for heating, which is fed with hydrocarbon or a similar fuel and responds quickly to a thermostatic control. A receptacle of small capacity is adapted to receive a small wick or pad which projects into the chamber formed between inner and outer perforated tubes. A thermostatically controlled valve for controlling a main flow of fuel to the receptacle and a fuel conduit provided with fins or other heat dissipating device

are attached to the apparatus. A means is also provided to allow a small or bye-pass flow of fuel to pass to the receptacle when the main flow is cut off. Such a lamp is particularly suitable for use in a refrigerator. W. S. E. C.

637. Apparatus for Obtaining Light and Heat by the Combustion of Liquid Hydrocarbons. Scott-Snell Laboratories Ltd. and C. and E. Scott-Snell. E.P. 342,749, 26.2.31. Appl., 23.5.30.

A burner for obtaining light or heat embodies a wick carrier provided with one or more notches which can be moved up or down vertically so as to adjust the level of the wick in relation to a vapour throat, and fixed shelves mounted at the lower end of the vapour throat. Air or oxygen is injected under pressure for admixture with the products of combustion of the initial flame.

W. S. E. C.

638. Blue-Flame Oil Burners. J. Imber. E.P. 343,396, 19.2.31. Appl., 6.5.30.

This invention relates to blue-flame burners of the kind wherein the upper end of an annular wick extends into a burner cone and a perforated flame spreader is disposed within the open upper end of the wick or inner wick tube. The improvement in the oil burner as claimed in E.P. 326,821 consists of an inner or lower cone provided with a series of perforations for the purposes of admitting an additional supply of air to the annular chamber.

W. S. E. C.

See also Abstract No. 670.

Lubricants and Wax.

639. Recovery of Lubricating Oils. T. Salomon. *Ann. des Comb. Liquides*, 1930, **5**, 831–843.

The deterioration of lubricating oils is due to various factors, such as the following: contamination with dust and metal particles (particularly when running-in new machines), water (from turbines or faulty cooling systems), volatile diluents (residues from incomplete combustion of gasoline or gas oil), the products of oxidation of the oil (acids, complex bodies soluble in the oil and sludge) and products of cracking in internal combustion engines (carbon and low-boiling products). The deterioration is accelerated by aeration and the catalytic action of metals.

For oils which are only slightly contaminated a process of purification may be used. Solids (metal particles sludge and carbon) may be removed by filtration or centrifuging. This is best carried out at as low a temperature as possible (45° C. to 100° C., according to the viscosity of the oil) in order to avoid further deterioration and the solution of sludge, which may re-separate on cooling. Colloidally suspended carbon is not removed by this treatment. The use of filtering-earth or reagents such as soaps, resinates or sulphoricinates may be necessary. Soaps are inadvisable, especially for turbine oils, as they promote deterioration and emulsification. The DMS ultra-filter or the Sapra electro-capillary filter may also be used. Water may be removed by centrifuging. For unstable emulsions water-washing as well is advantageous. For very stiff emulsions more drastic refining treatment is usually necessary. Diluents from gasoline or gas-oil residues may be removed by distillation with steam or vacuum.

When large amounts of chemical alteration products are present, a more thorough process of regeneration is required. This cannot always be carried out by the user and requires experienced workers. Treatment with alkalies,

silicates, etc., followed by earth-treatments, extraction with solvents such as glacial acetic acid or acetone, or re-refining with sulphuric or hydrochloric acids are among the suggested means of carrying out this refining process.

The purification of slightly used oils may be carried out continuously in the case of machinery using large quantities of oil, such as turbines or groups of Diesel engines, the oil passing through the purifier and returning to use; or the oil may be recovered in batches when sufficient used oil has accumulated. E. B. E.

640. Possible Synthetic Lubricating Oils. A. R. Bowen. *Oil and Gas J.*, 1931, **29** (42), 209.

An outline is given of the possibilities of the production of lubricating oils by (*a*) catalytic hydrogenation of low-grade lubricating oils, (*b*) low-temperature carbonisation and hydrogenation, with and without a catalyst, of coal and coal tars, and (*c*) the polymerisation or condensation with middle oils of olefines. In view of the complex processes involved, production of such oils can only be justified by their possessing a high degree of lubricating quality, as compared with normal petroleum oils. The criteria for the evaluation of such oils, *e.g.*, flat viscosity curve, low friction value, absence of impurities such as acidic and phenolic bodies, stability towards oxidation is given.

(*a*) The process which has been developed to the highest extent is the hydrogenation of low-grade petroleum oils (ref. J.I.P.T. 1931, **17**, 121). Such hydrogenated oils resemble high-class petroleum lubricants very closely.

(*b*) Oils produced by low-temperature carbonisation are generally characterised by instability towards oxidation, steep viscosity curves, high specific gravities, and usually contain quantities of wax, phenolic and nitrogenous compounds. Oils obtained by high vacuum distillation of German brown coals have been put on the market, but are stated to be definitely inferior in lubricating value.

A high yield of heavy oils is obtained by the Berginisation of coal, but there is not much data available on the properties of the fractions of lubricating-oil boiling range. The products from the hydrogenation of pure aromatic hydrocarbons are relatively stable viscous oils of high specific gravity and do not resemble the normal constituents of petroleum oils.

Viscous hydrocarbons, as well as light spirits, are formed from coal and coal tar in the catalytic hydrogenation process developed by the I. G. Farbenind., and are probably similar to those obtained from non-catalytic processes, though they may be more saturated.

(*c*) Ethylene can be readily polymerised under pressure in the presence of catalysts such as aluminium chloride or boron trifluoride, the oils obtained at ordinary temperatures consisting of a free oil which is viscous and mainly naphthenic in character and a complex aluminium chloride-hydrocarbon oil, from which an olefinic oil can be separated. At higher temperatures (100–180° C.) lighter oils are obtained, including spirits of paraffinous nature and lubricant fractions probably of polynuclear naphthenic structure. These oils are superficially similar to ordinary petroleum oils, but possess steeper viscosity curves and have a greater tendency to thicken on oxidation without, however, forming asphaltenes. Friction values on the Deeley machine compare favourably with those of well refined petroleum oils.

A fluid middle-oil from coal tar can be converted to a highly viscous lubricating oil by polymerisation and condensation with olefines in the presence of a catalyst. Little data is available concerning the nature of such oils, but they are probably similar to those formed by straight polymerisation of ethylene.

Indications are given of the lines along which further research is needed for the production of the most suitable grade of oil from the above processes. C. L. G.

641. Practical Analysis of Lubricating Greases. S. J. Caplan. *Petr. Eng.*, 1931, **2** (6), 104.

The analysis of those greases made from soaps and mineral oil only is considered from the point of view of duplicating samples tested. A centrifugal machine capable of rotating four aluminium cups weighing approx. 500 g. each at 1000 r.p.m. is required. The cups should be 8 in. tall, have a capacity of 400 c.c., be fitted with snugly-fitting caps and not vary by more than ½ g. in weight. This enables duplicate tests to be made on two samples simultaneously. 100 g. of grease is introduced into each cup and worked with sufficient naphtha of max. F.B.P. 210° F. to cause the grease to flow readily. More naphtha is added to balance weight of cups and fill to three-quarter capacity. The caps are then fitted and the cups rotated at 800 r.p.m. until the soap adheres to the bottom, when the naphtha extract is transferred to weighed distillation flasks. The process is twice repeated to ensure complete extraction of oil. The naphtha is removed from the extract by distillation, followed by blowing with inert gas, and heating to constant weight at an oven temperature of 212° F. If the oil so obtained contains more than ½% of unsaponified fat, this should be extracted and identified by the usual methods. The physical properties of the mineral oil and the percentage present can thus be determined. The cup containing soap is then heated to constant weight at 212° F., thus giving the total percentage of soap, filler and glycerine present.

The water content deduced by difference should be checked by a direct distillation test on the grease. A portion of the dried soap is ashed in order to ascertain whether the grease is loaded and the nature of the metallic base used. The usual methods of analysis are employed to determine the nature of the fatty acids present in the grease. R. A. E.

642. Preparation of Odourless Paraffin Wax. H. Wischnowitzer. *Erdöl und Teer*, 1931, **7**, 142–144.

Many attempts have been made to prepare odourless and tasteless paraffin wax, but these have generally proved unsuccessful.

No process is given in the literature for the preparation of odourless wax from petroleum, but Graefe gives the following for the preparation from brown coal tar. The wax is heated in a retort to 150° C. and blown for a long time with steam, which separates the odourous oil. Besides a smell due to the benzine used in extraction, paraffin wax has another unpleasant odour. It was at first thought that the contained oil was entirely responsible for this, as waxes containing more than 3% of oil, as estimated by Holde's method, could not be deodorised. That this was not the case was proved by sweating off 40% of a wax containing originally only 2·5% of oil. Treatment with sulphuric acid and earth only slightly improved the smell. Another supposition was that only waxes with a small melting range could be made odourless, *e.g.*, Scotch (range 3°) and Rangoon wax (range 2·8°). An apparatus for fractional sweating is described and a number of waxes, *e.g.*, Boryslaw (ranges 5·5°, 8°, etc.), were separated into fractions with ranges of 0·7, 1·5°, etc. (Solidifying pt. of fractions were determined by Shukoff's method.)

The characteristic smell of these narrow fractions could not be removed by treatment with 66° Bé. acid and earth. The smell was not due entirely to oil produced in preparing waxes by cracking distillation, since complete removal of the oil still left the odour.

The effect of different refining processes was then examined, various treatments with 66° Bé. acid, fuming acid, filtering earth and air-blowing being investigated. The most efficient and economical process was found to be the following: the crude paraffin wax was treated with 4% of 66° Bé. acid, then

with 2% fuming acid, washed with sodium carbonate and then treated with a mixture of 1·5% "Florida-earth" and 2% "Montana." This process deodorised waxes either of narrow or wide range.

Treatment to remove odour has little effect on either refractive index or solidifying point of the fraction. Losses on treatment vary from about 1·7 to 5·8%, and are greater with the higher melting point fractions and with the use of fuming sulphuric acid. E. B. E.

643. Manufacture of Lubricants. W. B. D. Penninan. E.P. 317,406, 16.2.31. Appl., 15.8.29. Conv. (U.S.A.), 15.8.28.

The lubricants consist of mineral oils to which has been added a polar body, a material to produce "mushiness" and a de-oxidant. The polar bodies may be saponifiable oils, fatty acids or higher alcohols and ketones. "Mushiness" may be produced by the addition of mixtures of diphenylamine and tallow oil, soap solutions or sulphonic acids. Lead tetraethyl, phenol disulphide or a number of other compounds may be used as de-oxidants. With these additions a lubricant for heavy machinery may be made from oil of low viscosity (below 135 Saybolt at 100° F.). T. A. S.

644. Cleansing of Oils. J. F. Stallard. E.P. 342,400, 5.2.31. Appl., 11.11.29.

Used lubricating oil is purified by spraying on to the surface of the oil a solution containing 4 oz. of caustic soda, 4 oz. of sodium silicate and 4 oz. of talc powder in 8 oz. of water. The solution is maintained at 17° and circulated by pumping from the bottom of the vessel in which the treatment is being carried out to the spray above the oil. The impurities collect on the surface of the refining liquid. T. A. S.

645. Increasing the Viscosity of Hydrocarbons, Oils and the Like. T. M. Rigby. E.P. 343,548, 16.2.31. Appl., 16.11.29.

The viscosity of lubricants is increased and permanent emulsions for use as fuel are obtained by adding rubber latex to oils and wholly or partially coagulating the rubber by suitable chemical treatment. T. A. S.

646. Separation of Paraffin from Petroleum Oils and Apparatus Therefor. J. Verner, Assr. to Standard Oil Dev. Co. U.S.P. 1,789,347, 20.1.31. Appl., 16.8.27.

Paraffin in suspension in paraffin oils is separated by forcing a mixture of the cooled oil under pressure through a combined filter and sweating zone. The oil is forced through an inlet to a vertical shell having top and bottom closures. The case is provided with a large number of spaced canvas filtering thimbles closed at the upper end and containing tubes which can be heated. The oil flows through these thimbles and through perforated sleeves beneath the thimbles. A series of spaced horizontal grid-like partitions are provided adjacent to the filtering medium to help to support the paraffin layer. The oil is withdrawn as produced through a valved outlet in the bottom closure. The temperature is then increased until a relatively pure paraffin is left in the filtering medium (100–130° F.) and further increased to melt the paraffin which is withdrawn separately. W. S. E. C.

647. Oil Purification Process. F. M. Clark and A. T. Harding, Assrs. to General Electric Co. U.S.P. 1,798,968, 31.3.31 Appl., 29.10.28.

This purification process is applicable to the refining of crude or used mineral lubricating and insulating oils. The process consists in bringing the oil to be purified into intimate contact with a solvent for the impurities, which is a good dielectric, chemically neutral and substantially insoluble in the oil. The

preferred solvent used for extracting the impurities is tricresyl phosphate. The removal of dissolved tricresyl phosphate from the oil is accomplished by addition of acetic acid. After separation the purified oil is freed from traces of the reagents by neutralisation and redistillation. The automatic purification of insulating oil during use may be effected by addition of tricresyl phosphate to the unused oil; as oxidation proceeds the ester becomes insoluble and separates, carrying with it the dissolved oxidation products.

R. W. L. C.

648. Treating Petroleum Oils. H. B. Setzler and M. C. McDonald, Assrs. to National Refining Co. U.S.P. 1,791,329, 3.2.31. Appl., 27.10.21.

Petroleum oils containing amorphous wax are cooled to 40° F. and agitated while chilled with sulphuric acid. The oil and acid are agitated to precipitate the sludge and amorphous wax and the oil and semi-solid constituents are separated.

W. S. E. C.

Fuel Oil.

649. Burners for Heavy Oils and the like Fuels. A. Lanser. E.P. 340,858, 8.1.31. Appl., 27.2.30.

The end of the nozzle is constructed and given an ogival form and a second nozzle is arranged inside the first and is of such dimensions that there is a clear space between the two.

The nozzle of the atomiser is fitted with a conical deflector. This is provided with a number of tubular projections, the axes of which are tangential to the atomiser, and also with at least two series of cylindrical holes.

The atomiser is arranged in the mouthpiece so that the large base of the deflector cone is at a distance from the mouth equal to the internal diameter of the mouthpiece in the vertical line of this base.

The end of the atomiser is so designed that it forms a dripper, and any oil falling from this is collected in a small tube and delivered outside the burner.

W. L.

650. Separation of Mineral Oil from Emulsions. F. G. Edmed and H. Newington. E.P. 343,487, 16.2.31. Appl., 16.11.29.

Emulsions, such as the fuel residues from ships' tanks, are broken up by the addition of tar or creosote oils or phenols. The mixture is agitated in a tank and may be heated up to 65°.

T. A. S.

651. Burner for Oil Fuel. V. T. Standring, N. C. Christie and R. W. Gray. E.P. 343,923, 25.2.31. Appl., 25.11.29. Conv. (N.Z.), 27.11.28.

A description is given of an oil fuel burner for use in the furnaces of steam boilers wherein a cylindrical plug is removably fitted into a casing, the support being given by the engagement of the casing wall with the peripheral surface of the plug. The latter is formed with five spiral passages leading into an obtuse-angled conical chamber between the outer end of the casing and the corresponding end of the plug which terminates the chamber without projecting thereinto. The outer end of the casing is provided with a central countersunk bore communicating with the chamber.

W. H. T.

Fluxes and Asphalts.

652. Accelerated Tests of Asphalts. O. G. Strieter. *Bur. Standards J. Res.*, 1930, **5**, 247–253.

Equipment and methods for testing asphalt by accelerated weathering are described.

The equipment consists of three carbon arc lamps (220 volts D.C. and 13 amps.). Each lamp is fitted with a cylinder with 3 in. slots for holding the test panels and each cylinder is geared to a driving mechanism which rotates it once in 20 min. Light spray is obtained from vertical water jets and lawn sprinklers with rotating arms are used for rigorous spraying. The lamps maintain a temperature of approximately 140° F. at the surface of the panels and a refrigerator, as a separate unit, is used to obtain sudden changes of temperature.

The test panels used were 0·025 ± ·003 in. thick and were backed with an aluminium panel.

It was found that asphalts show a characteristic behaviour when exposed to the accelerated cycle so that it is possible to classify the asphalts into "types of behaviour."

It was also found that some grades of asphalt contract when exposed to the cycle as well as outdoors.

The following cycle is put forward as a tentative standard of test: cold 1¾ hrs., rain 1 hr., light 1½ hrs., rain 2 hrs., light 16¼ hrs.

Chemical analysis was found useful in following the progress of deterioration. The oil and resin content was found to decrease on weathering and the asphaltene content to increase. W. L.

653. Pitch Compositions. Barrett Co. E.P. 343,265, 19.2.31. Appl., 13.1.30.

This invention relates to a pitch composition especially adapted for use as a protective coating for surfaces which are exposed to weathering, such as those of pipes, lines, roofs, etc. The composition comprises pitch and mica and has a m. pt. higher than the pitch (above approx. 125° F.). The mixture contains a major proportion of pitch and from 10–30% of finely-divided mica (60–100 mesh) homogenously admixed therewith, the composition being freely fluid at temperatures somewhat above its m. pt. W. H. T.

654. Production of Aqueous Dispersions of Bituminous Materials. H. D. Elkington. E.P. 343,427, 9.2.31. Appl., 9.10.29.

A process is described for the manufacture of aqueous dispersions of bituminous materials such as asphalt, pitch and the like, which possess a high degree of suspendability of the dispersed particles.

The bituminous material is dispersed with a dispersing agent such as bentonite or a mixture of argillaceous materials such as bentonite and clay with or without the addition of protective colloids. The amount of the dispersing agent used must not exceed 10%, calculated on the weight of the dispersed phase and the operation is effected at a temperature not substantially higher than the m. pt. of the substance to be dispersed. The final product so produced has a water content of 30–40% and an average size of particle of 10 microns. The relatively thick dispersion discharged from the dispersion apparatus is then beaten or whipped without the addition of water.

W. S. E. C.

655. Treatment of Bituminous Street Coverings. Continentale Teerstrassenban. G.m.b.H. and H. Köhler. E.P. 343,692, 26.2.31. Appl., 15.1.30.

Depressions which are formed in the bituminous covering layer of streets, especially asphalt streets, *e.g.*, holes, channels of rectangular, square or semi-circular cross-section formed in straight lines, etc., are filled either by surface-tarring with coal tar or by introducing tar-sand, tar chippings or tar concrete. W. S. E. C.

656. Reclaiming Asphalt from Residuum. R. P. Crothers. U.S.P. 1,788,792. 13.1.31. Appl., 27.3.28.

Residuum oil from a cracking plant is mixed with fuel oil and the two are warmed and caused to flux. The mixture is then warmed and maintained in a fluid condition without any appreciable agitation so as to allow the free carbon to settle. The fluid is withdrawn from the top and any remaining free carbon may be separated either by further heating and settling or by centrifugal action, while in a hot and fluid state.

The withdrawn asphalt is subsequently treated, preferably by heating to reduce it to a commercial asphalt. W. L.

Special Products.

657. Solid Carbon Monoxide from Mexico. J. W. Martin. *Ind. Eng. Chem.*, 1931, **23**, 256–258.

On the Quebrache oilfield, south of Tampico, large quantities of carbon dioxide issue with the crude oil—the gas containing about 95% carbon dioxide and 5% combustible gases. A process has been worked out on a semi-commercial scale for producing pure solid carbon dioxide which can be economically shipped to New York. C. C.

658. Hydrogenation of Pyridine with Hydrogen under Pressure by the Bergius Process. H. Thate. *Rec. Trav. Chim.*, 1931, **50**, 77–90.

The products obtained by hydrogenation of pyridine at 500°/50–100 atm. have been investigated qualitatively and quantitatively. In the absence of a catalyst absorption of hydrogen begins at 450°. Analysis of the gaseous products after removal of ammonia shows the presence of H (67·9%), CO (1·2%), N (1·2%), methane (13·8%), ethane (7·4%), propane (4·7%), butane (2·0%), butane + pentane (1·0%), ethylene + propylene (0·5%) and propylene + butylene (0·4%), the hydrocarbons being formed by cracking of the pentane originally formed. Steam distillation from an acid solution of the liquid products (1·2%) yields pentane, valeronitrile, an unsaturated hydrocarbon $C_{16}H_{14}$ (formed only when 1% of aluminium or ferric chloride is added as catalyst), and nitriles, b. pt. 220–260°. Steam distillation from an alkaline medium affords primary amines (containing n-amylamine), unchanged pyridine, 3 and 3 : 5 alkylated piperidines, 2 : 2^1 dipyridyl, and, probably 2 : 4 dipyridyl. The solid product consists of carbon and ammonium cyanide. The relative proportions of (1) hydrocarbons, (2) nitriles, and (3) secondary and tertiary amines formed vary with the conditions of hydrogenation. Thus at 500°/50 atm. for one and four hours proportions are, respectively, (1) 16, (2) 4, (3), 2·5% : and (1) 29, (2) + (3), 2·5%, whilst at 500°/100 atm. for one hour they are (1) 25%, (2) 4·6% and (3) traces only.

Since similar products are obtained by hydrogenation of piperidine, the mechanism of the hydrogenation of pyridine is considered to be primarily reduction to piperidine, which subsequently breaks down to the products

enumerated above, and a scheme of the formation of these is given. A summary of investigations on the hydrogenation of both aromatic and aliphatic compounds is given. W. S. E. C.

659. Formation of Methyl Alcohol by the Direct Oxidation of Methane. W. A. Bone. *Nature*, 1931, **127**, 481.

Substantial amounts of methyl alcohol have been obtained by Newitt and Haffner by the direct interaction of oxygen and methane at 360° C. and a pressure of 100 atm. Using methane and oxygen in ratio 9 : 1, the action was complete in a few minutes, 17% of the methane burnt being recovered as methyl alcohol, 0·6% as formaldehyde and the remainder as oxides of carbon and steam. No hydrogen or peroxide was produced. This shows that the slow oxidation of methane proceeds throughout in accordance with the hydroxylation theory. C. C.

660. Manufacture of Aliphatic Acids and Esters. H. Dreyfus. E.P. 340,939, 1.1.31. Appl., 1.10.29.

Aliphatic acids are manufactured either in the free state or in the form of their esters by causing alkyl ethers to react with carbon monoxide in the presence of one or more non-acidic catalysts known to promote this particular synthesis. These catalysts are substances which are, or are capable of forming, acetates which split off acetic acid at temperatures under about 400–450° C. and preferably between 200° and 300° C. One or more catalysts may be employed and the following examples are quoted: copper, tin, lead and zinc oxides, copper and zinc acetates, zinc, aluminium and tin methylates. Basic materials, such as potassium or sodium acetates, may be admixed with one or more of the above catalysts. The reaction is performed under pressure, *e.g.*, from 50 to 300 atm. and at temperatures between 200° and 450° C. The reaction products, whilst still in the vapour state, may be saponified to produce aliphatic acids of high concentration (see Spec. No. 284,582). Examples are given of the production of methyl acetate and acetic acid from dimethyl ether. W. D. S.

661. Manufacture of Aliphatic Acids. H. Dreyfus. E.P. 341,031, 6.1.31. Appl., 6.9.29.

Aliphatic acids are manufactured by subjecting the vapours of aliphatic alcohols to the action of oxygen or a gas containing oxygen. The operation is performed at temperatures of between about 150° and 400° C. and in the presence of a catalyst comprising one or more metallic oxides, hydroxides or carbonates of the alkali or earth alkali metals. The temperature must be insufficiently high to cause the formation of substantial quantities of acetone or other ketones, and it is inadvisable to use temperatures exceeding 300° C. It is preferable to perform the reaction in the presence of water vapour to facilitate the decomposition of aliphatic acid salts to free acid. An example is given of the formation of acetic acid by passing a mixture of ethyl alcohol, oxygen and steam in about the proportions of 1 : 3 : 5 by volume over ferric oxide maintained at 280–320° C. If a mixture of calcium carbonate and ferric oxide is used as a catalyst lower temperatures, *e.g.*, about 230–280° C., should be maintained. W. D. S.

662. Manufacture of Hydrogen or Hydrogen-Containing Gases from Methane and Similar Hydrocarbons. D. Tyrer and I.C.I., Ltd. E.P. 341,462, 5.1.31. Appl., 3.10.29.

A generator is surrounded for part of its height by a jacket and the carbon monoxide produced during the blowing period is burnt in the jacket with the aid of extra air, the heat produced being transmitted to the coke in the

generator. Hydrocarbon gas is then admitted through the jacket where it is preheated, and then upwards through the coke bed, where it is decomposed into hydrogen and carbon, the latter being deposited on the coke. This carbon is burnt during the "blow" period and supplies sufficient heat for the process if pure hydrocarbon gases are used. If the hydrocarbon gases are diluted with inert gas or steam it is necessary to add coke. T. A. S.

663. Production of Alkylene Derivatives from Alkylene Oxides. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 342,022, 29.1.31. Appl., 11.11.29.

Hydroxy-alkyl compounds are produced by passing an alkylene oxide together with water continuously through a heated reaction vessel. In order to effect the reaction in the absence of catalyst it is necessary to work at a pressure above atmospheric. Organic hydroxyl or carboxyl compounds may be substituted for water, but in either case it is desirable to carry out the reaction with a large excess of diluent in order to prevent the formation of high boiling byproducts. The latter are formed in a greater or less degree by the introduction of further molecules of alkylene oxide on to the already formed hydroxy-alkyl compounds. The resulting mixture is then freed from water or from the hydroxyl or carboxyl compound, leaving polyhydric alcohols, their esters or ethers. Examples are given of the formation of ethylene glycol monoethyl ether from ethylene oxide and ethyl alcohol reacting at a temperature of 180° C. and a pressure of about 30 atm. of propylene glycol monoethyl ether from the corresponding oxide under similar conditions, and of other ethers and esters. W. D. S.

664. Polymerising Hydrocarbons of the Butadiene Series. I. G. Farbenind. A.-G. E.P. 342,107, 29.1.31. Appl., 9.1.30.

Hydrocarbons of the butadiene series are polymerised by the use of sodium powder which has previously been brought into a finely homogenous distribution in an indifferent solid diluent such as a metallic salt. This expedient produces a very uniform course of the reaction and the very uniform polymerised products thus obtained constitute a caoutchouc of high value. About 0·5%, of the quantity of hydrocarbon of sodium is used. The optimum temperature range lies between 35° C. and 45° C. W. D. S.

665. Production of Acetylene. J. Ferguson and I.C.I., Ltd. E.P. 342,771, 9.2.31. Appl., 8.11.29.

It is claimed that acetylene can be obtained in good yield from hydrocarbons other than methane, especially olefines, by thermal treatment in the presence of an excess of steam, using temperatures over 1000° C. and short contact times. Temperatures between 1200° C. and 1750° C. and heating times up to 2 sec. are suggested. With methane temperatures above 1400° C. are necessary before acetylene is produced in any quantity. The quantity of steam required increases with the number of carbon atoms in the hydrocarbon under consideration, thus for ethylene a five-fold excess of steam is suitable, but usually the range is 5 to 20 times the volume of hydrocarbon when higher olefines are present. Any carbon formed during operation can be removed by steaming or air-blowing. Examples are given. E. N. H.

666. Catalytic Gaseous Reactions. J. L. Brill. E.P. 342,854, 12.2.31. Appl., 31.12.29.

The process indicated relates particularly to catalytic exothermic gaseous reactions and the methods and apparatus for initiating reactions which are to be carried out at elevated temperatures and pressures. In order to start a

reaction a priming catalyst at a relatively low temperature is used and the gases so heated are then directed upon the catalyst for the main reaction. In this way it is possible to avoid trouble due to high temperatures in high pressure pipes outside the actual reaction apparatus.

Examples relating to the synthesis of methanol and higher homologues of methanol are given. E. N. H.

667. Manufacture of Mercaptobenzothiazoles. Goodyear Tyre and Rubber Co. E.P. 343,013, 9.2.31. Appl., 8.8.29.

During recent years mercaptothiazoles and various derivatives thereof have become highly important as vulcanisation accelerators in rubber compounds. An improved process for preparing such compounds, mercaptobenzothiazole or nuclear substituted derivatives is described. It comprises treating an alkali salt of *o*-amidothiophenol, or a nuclear substituted derivative thereof, with an acid other than hydrogen sulphide, such as hydrochloric or sulphuric acid, in the presence of carbon bisulphide. W. D. S.

668. Production of Oxidation Products from Crude Paraffin Hydrocarbons, Waxes and the Like. M. Luther and E. Willfroth, Assrs. to I. G. Farbenind. A.-G. U.S.P. 1,788,799, 13.1.31. Appl., 8.4.29.

In the process described oxidation products from crude paraffin hydrocarbons and waxes are produced by acting upon the initial substances in the liquid state with gases containing oxygen and nitric oxides in the presence of filler bodies with a large superficial area and of a metal enolate. W. S. E. C.

See also Abstract No. 640.

Refining and Refinery Plant.

669. Action of Sulphuric Acid in the Refining of Gas-Benzine and Tar Oils from Brown Coal. S. Ruhemann. *Erdöl und Teer*, 1931, 7, 123–126.

The oils from brown coal consist of a very complicated mixture of aliphatic and cyclic hydrocarbons, together with oxygen and sulphur compounds, and include some characteristically unstable compounds.

The materials used in this work were a gas-benzine and a generator-tar, which were first washed with 5% acid and 10% alkali, and then fractionated. It was found that treating the oil with hydroferrocyanic acid effected an incomplete removal of oxygen compounds, including ketones, esters and alcohols, which could be recovered by treating the extract with alkali.

The neutral oil was then treated with sulphuric acid. A marked reaction set in with the gas-benzine with 60% acid, but not with the generator-tar until 80% concentration was reached. The gas-benzine contains a large amount of gum-forming constituents which resinify on first treating with acid. The less gum is formed, the more the oil has previously been treated with hydroferrocyanic acid.

On diluting with water the acid layer separating after shaking with diluted acid, an oil separates out. This has approximately the same boiling range as the original fraction and has a high oxygen and fairly high sulphur content. This oil darkens rapidly and its smell resembles that of aromatic ketones.

The oil isolated in this way from generator-tar oil contains only a small amount of alcohols and small quantities of ketones such as acetophenone and a ketone $C_7H_{14}O$. By the action of hydroferrocyanic acid ethers, *e.g.*, of the formula $C_{10}H_{14}O$, were isolated.

The sulphur compounds isolated from the acid-soluble oil are thought to be present in the original material as sulphides and thiophene. Allyl sulphide was found to dissolve in 80% sulphuric acid and to be recovered unchanged on dilution. The effect is probably due to the formation of easily hydrolisable sulphonium salts. The oxygen compounds are, analogously, probably present in the acid-soluble oil as oxonium salts.

From the action of 80% acid, the formation of alcohols and sulphuric esters was expected. Alcohols were not formed in the case of the generator-tar oils, but the presence of mono- and di-sulphuric esters was demonstrated in the gas-benzine fraction. These esters are formed from aliphatic olefines, *e.g.*, nonylene, which were shown to be present in the gas-benzine fraction.

High-boiling polymers were not observed to be formed from the tar fraction until 96% acid was employed, but 60% acid gave these products with gas-benzine. These polymers have the general formula $C_n H_{2n-6}$ (*e.g.*, $C_{18} H_{30}$, $C_{24} H_{42}$) except in one case, when the type $C_n H_{2n-10}$ (*e.g.*, $C_{24} H_{38}$) was shown to be present. By the action of bromine on these compounds, hydrobromic acid was split off. Oxidation with chromic acid or permanganate gave fatty acids, principally acetic or oxalic acids. They will not hydrogenate with a nickel catalyst, but crack on raising the hydrogenation temperature. They could, however, be depolymerised by the action of floridin at about 250°, giving cyclic compounds with two or sometimes three double bonds. Condensations of the type found by Spilker, between substituted benzenes and styrene, may be involved.

The well-known rise of sulphur-content on intensive treatment with sulphuric acid was observed in the case of the repeated solution of undecylene in acid and its regeneration by thermal decomposition, and is apparently due to the formation of sulphides.

The tar from brown coal also contains aromatic compounds which give sulphonic acids with excess of concentrated sulphuric acid. After treatment with acid containing 8% of anhydride, the residue from gas-benzine is of a paraffinic nature. It is not attacked by nitric acid and will not dehydrogenate with nickel. The residue after treating generator-tar with 96% acid still contains unsaturated cyclic compounds.

The recovered products from sulphuric acid refining of brown-coal tar are suitable, by reason of their great stability, for use as high-grade lubricating oil. E. B. E.

670. Refining by Continuous Counter-Current Treatment. Anglo-Persian Oil Co. (Chem. Development Staff). *Oil and Gas J.*, 1931, **29** (42), 441.

The advantages of a continuous counter-current treatment of oils over batch washing are outlined and descriptions given of the main types of previous systems of continuous counter-current washing as compared with the new method developed by the A.P.O. Co.

Continuous treatment results in the production of a better refined oil and usually in saving time and reagents due to the more complete utilisation of the reagents, the nearly spent reagents being applied to the raw oil and the active reagent to the nearly finished oil, whereas in batch treatment the activity of the reagent decreases with the amount of treatment the oil has undergone. Labour, fires risks and breathing losses are also lessened.

Previous plants were either of the tower type, pump agitated vessels or orifice mixer tanks. In each of these the time of contact is short. Control of flow and composition of mixture is difficult.

Improvements can be effected in the tower type by the use of a central staff fitted with paddles, but this is only practicable for small towers, while increasing the size and number of pumps in the other two types would be expensive.

The plant introduced by the A.P.O. Co. is used for the treatment of straight-run and cracked benzines and heavier distillates, including heavy kerosine with either alkaline hypochlorite, acid or alkali.

It consists of a series of pairs of vessels, one of which is an agitator and the other a separator, the oil being metered in at one end of the series and the reagent at the other. The oil is thus alternately agitated with and separated from the reagent, which becomes progressively stronger from pair to pair. Movement from vessel to vessel is made by taking advantage of the difference in density of the oil reagent and emulsified mixture. The mixing vessels are provided with efficient paddle agitators and valves are provided for the regulation of the desired proportions of oil to reagent. A description and flow sheet is given. C. L. G.

671. Sulphur Compounds in Light Oils. F. C. Parris. *Oil and Gas J.*, 1931, **29** (42), 148.

The amount of refining treatment given to light oils usually depends to a very large extent on the amount and type of sulphur compounds present in the oils. The most economical and effective treatment can only be decided upon after a full investigation of the types of sulphur compounds occurring in the different products from the crude. The chief types of sulphur compounds occurring in light oils are: Hydrogen sulphide H_2S, mercaptans R-S-H, alkyl sulphides R-S-R, disulphides (thioethers) R-S-S-R, thiophenes C_4H_4S, free sulphur S, sulphones $R\text{-}SO_2\text{-}R$, sulphonic acids $R\text{-}SO_2\text{-}OH$, sulphonides R-SO-R. The reaction of the different types of sulphur compounds towards refining agents is given.

Hydrogen sulphide is the most corrosive and toxic of these compounds and both occurs free in crudes and is formed by decomposition of higher sulphur compounds. It should be removed at as early a stage as possible in the refinery operations, both on account of its dangerous nature and because of its reaction towards sulphuric acid during later refining treatments, when free sulphur is formed which dissolves in the oil. It can be removed by weak caustic soda, sodium carbonate or ammonia.

The mercaptans are liquids and gases of intolerable odour and are insoluble in water. The lower mercaptans are soluble in caustic alkali solutions and form crystallisable mercury mercaptides with definite melting points, and lead mercaptides of yellow colour. They react more readily with plumbite or acid than the higher mercaptans. The latter, although having less corrosive tendencies than the lower mercaptans, still give positive doctor tests. Mercaptans are converted to disulphides by sulphuric acid plumbite solution, sodium hypochlorite, etc. These do not show a positive doctor test and have not an unpleasant odour, but are relatively insoluble in aqueous solutions (particularly the higher homologues) so that they go back into solution. Hence the mercaptan sulphur content is not reduced. The disulphides are only slightly soluble in sulphuric acid, but form addition products with aluminium chloride.

Alkyl sulphides possess strong odours and are insoluble in caustic alkali solutions. On oxidation they form sulphides and finally sulphones. Addition products are formed with halogens, mercuric chloride and alkyl iodide. Alkyl sulphides are more or less soluble in acid but are not affected by sodium hypochlorite or copper oxide at ordinary temperatures. The sulphones formed are stable, odourless and neutral compounds.

Sulphonic acids are formed by oxidation of mercaptans and are soluble in water and acids. They can be removed by alkali and react with nitric acid and ammonia, giving nitrates and amines.

Thiophenes are not affected by doctor solution, copper oxide or sodium hypochlorite, but the lower members can be removed by sulphuric acid, yielding sulphonic acids.

Free sulphur can be partly removed by absorption in lower mercaptans, or washing with concentrated sodium sulphide solution. C. L. G.

672. Combating Corrosion with Ammonia. E. R. Woodward. *Oil and Gas J.*, 1931, **29** (42), 139.

The increasing interest in the problem of combating corrosion of refinery equipment is chiefly due to the increased amount of sour crudes which are now worked up. Such crudes are usually cheaper than sweet crudes and yield gasolines with higher anti-knock values. Corrosion to refinery equipment is calculated to cost $16.70 per year for every barrel per day of plant capacity, also causing additional shut-down periods and fire and other hazards to operators.

Corrosion of different types is met with under (*a*) low temperature conditions and (*b*) high temperature conditions.

The corrosion of condensers and vapour lines is usually due to the HCl formed by the hydrolysis of the MgCl contained in the brine associated with the crude. Sodium chloride itself is not corrosive. Chromium iron and chromium nickel steels have not been found satisfactory for resisting this type of corrosion, the addition of ammonia being the most satisfactory preventative. The most convenient method is by the introduction of anhydrous ammonia from a cylinder, by means of an inlet pipe into the top of the bubble tower and into the vapour line. The outlet of the cylinder is raised to ensure that only gas is passed out, a trap acid check valve being desirable to prevent liquid issuing. The quantity of ammonia added is adjusted by a control valve so as to maintain the water coming over into the receiving house only just alkaline. Too rapid a flow of ammonia from the cylinder can be detected by the formation of ice on the outside of the cylinder. If the flow exceeds 25 lb./day ice will be formed, but if a larger quantity is required, two or more cylinders should be manifolded. The amount of ammonia required usually varies from 3 to 25 lb. per 1000 brl. crude. The effectiveness of the action of ammonia can be ensured by testing the water coming over for the presence of dissolved iron.

The increasing use of ammonia for combating corrosion has led refiners to consider its use from pressure tank-cars containing 50,000 lb. anhydrous ammonia, fitted with safety valves for 200 lb./sq. in. pressure. The anhydrous ammonia can then be passed into a tank containing water and stored as aqueous ammonia or stored in high pressure tanks in the gaseous state and piped to any point in the refinery.

It should be realised that ammonia is not suitable for combating the corrosion caused at high temperatures by sulphur compounds, owing to the low dissociation temperature of the sulphur compounds of ammonia formed. Hydrated lime used in proportion of ¼ lb. per lb. in the form of a slurry, and injected into the crude oil charging line, has made it possible to reduce corrosion almost entirely in the case of distilling Winkler County (W. Texas) crude. Hydrated lime is also used extensively to reduce corrosion in cracking plants. C. L. G.

673. Exchange of Heat: Heaters and Coolers. C. H. Butcher. *Ind. Chem.*, 1930, **6**, 404–407. Notes on Heaters, Including Air Heating by Steam. C. H. Butcher, *Ind. Chem.*, 1931, **7**, 95–96.

Various points in the design of plant for the exchange of heat are discussed. It is essential to fix the linear velocity of the fluids—either liquids or non-condensing gases—within reasonable limits, depending upon (*a*) cost of

pumping liquids or blowing gases, and (*b*) the best conditions for transference of heat. After fixing the fluid velocities, tubes of suitable diameter can be selected, and the number of tubes necessary to give the desired fluid velocity determined. Using coefficients of heat transference obtained preferably from data obtained from a similar plant in successful operation, the heat exchanging surface required can be calculated, and the necessary length of tube to give this surface determined.

Heat exchangers are divided into two main types: "tubular" and "double-pipe." For the former, the ratio of tube length to diameter is, in general practice about 100, and the diameter of the tubes 1 in. to 1¼ in. For "double-pipe heaters" 1¼ in. pipe inside 2 in. pipe is convenient. The disposition of the two liquids in a tubular heat-exchanging system is determined by the possible corrosion of the heat exchange surface, the liquid liable to cause the greatest corrosion, or give the worst deposits, being so placed that the surface with which it is in contact can be made accessible for cleaning. The closest convenient spacing of the tubes is with a pitch (centre to centre) equal to 1·5 times the external diameter of the tube. For this pitch the ratio of available cross-section inside the tubes is approximately 2 to 1. An example is given of the design of a tubular heater to perform a specified duty.

In designing tubular systems in which one or both fluids may be a non-condensing gas, it may be assumed that the non-condensing gases have the same heat transfer coefficients as air, since any small variation would be covered by the factor of safety introduced when computing the final overall coefficient. The following cases are considered.

Air Flowing Inside Pipes.—Weber's formula for calculating the heat transfer coefficient of any non-condensing gas with a molecular weight from 17–44, flowing in pipes of ½ in. to 2 in. bore, within the temperature range 70° to 2000° F. is given as follows:—

$$K = \frac{0{\cdot}88(V)^{0{\cdot}8}\,(T)^{0{\cdot}5}\,(S)^{0{\cdot}2}\,(Cp)}{(M)^{0{\cdot}3}}$$

where V is the mass velocity of the gas in lb. per second per sq. ft. of pipe cross-section, M the average molecular weight of the gas, Cp the average specific heat at constant pressure, T the arithmetic mean temperature in °F. absolute, S the surface factor. For air flowing inside pipes of 1 in. to 2 in. bore the coefficient is approximately equal to $fV^{0{\cdot}8}$, where f is a factor which varies from 3·7 to 4·5. For a pipe of 1 in. bore, where the air is at a temperature of 200° F., the value of "*f*" is 4·2.

Air Outside Pipes Parallel to Surface of Steam Pipes.—The heat transferred in B.Th.U. per hour per sq. ft. of heating surface per °F. mean temperature difference between the air and the surface of the steam pipes is equal to

$$\frac{2 + 5{\cdot}5\sqrt{v}}{4{\cdot}9}$$

where v is the linear velocity of the air in feet per second. The heating surface in sq. ft. (A) necessary for the transference of a number of B.Th.U. per hour, (Q), is then obtained by the formula:—

$$A = \frac{Q}{\Delta t_m\, k}$$

where k is the coefficient of transfer of heat and Δtm is the mean temperature difference. Higher heat transmissions are obtained in "double-pipe" heaters, and in both cases surface area efficiencies will increase as the diameters of the pipes decrease.

Air at Right Angles to Surface of Steam Pipes.—For tubular heaters of this type the heat transference in B.Th.U. per hour per sq. ft. per °F. mean temperature difference may be calculated by Carrier's formula :—

$$k = \frac{22{\cdot}3\,w}{w + 1{\cdot}42}$$

where w is the mass velocity of the air in lb. per sec. per sq. ft. of available cross-section at the centre line of one row of steam pipes.

Heat Transfer Coefficients for Steam and Water.—The heat transfer coefficient for vapours capable of complete condensation is given as well over 2000. Provision should be made for rapid and efficient removal of condensate. The overall coefficients used in designing tubular heaters operating on steam vary from 200–350 B.Th.U. per hour per sq. ft. per °F. mean temperature difference. The coefficient of heat transference when water is used as the cooling medium is $450v^{0.75}$, where v is the linear velocity of the water in feet per second. N. E. J.

674. Conditioning Refinery Water. S. Born. *Nat. Petr. News*, 1931, (6) 52–56 ; (7) 55–57 ; (8) 51–55 ; (9) 65–68 ; (10) 49–52.

The significance of various foreign substances is reviewed with reference to normal boiler-house practice and a complete scheme for rapid routine analysis of feed water drawn up. Chemical reactions involved in water softening are given in full and sources of hardness in water are discussed in light of appropriate methods for their removal.

The sodium/aluminium silicate and soda/lime type processes are discussed and described in some detail. Reduction of scale forming compounds and prevention of corrosion are dealt with in reference to cooler practice. H. G.

675. Modern Boiler Feed Water Treatment Methods. A. E. Warner. *Chem. Markets*, 1931, **28**, 279-283.

After drawing attention to the importance of the proper treatment of boiler feed water in the economical control of steam generation, and the value of routine chemical analyses to indicate the treatment required, a brief account is given of the advantages of the use of sodium-aluminate. Old methods in which compounds of unknown composition were used according to standard instructions were replaced by lime-soda external treating plants, the high initial cost and maintenance of which restricted their use. The introduction of sodium-aluminate in a usable form for treatment of boiler feed water was a decided improvement. Not only has sodium-aluminate proved successful in the direct treatment of boiler feed water, but it has also proved of use in speeding up the reactions in the lime-soda process. Acceleration is obtained, without adding hardness, owing to the alkaline nature of sodium-aluminate. N. E. J.

676. Vaporisation of Heavy Oils in the Manufacture of Oil Gas. C. Chilowsky. E.P. 342,447, 5.2.31. Appl., 4.12.29.

An emulsion of oil and water, with air, is atomised downwards into a vertical iron tube which is externally heated by means of the gas from a generator. The vertical tube is constructed of U-shaped members welded together, which gives a large heating surface. T. A. S.

677. Continuous Distillation Apparatus for the Refining of Oils and Fats and for Like Purposes. P. Pritchard and G. W. Lacey. E.P. 342,921, 12.2.31. Appl., 25.2.30.

The apparatus consists of a vertical cylindrical vessel built up of a number of superimposed similar cylindrical sections. The vessel is divided into a number of compartments by radial baffles, having staggered passage-ways

formed in sections corresponding in depth to the sections of the body. The oil is conducted in a tortuous path through the successive compartments, and steam or other gas is introduced through a number of small passages in one or more of the baffles adjacent to the bottom of the vessel. By this means the gas passes upwardly through the liquid and removes the volatile constituents through openings at the top of the vessel. W. S. E. C.

678. Separation of Oil from Bleaching Clay. W. W. Triggs from Deutsche Gasolin A.-G. E.P. 343,311, 19.2.31. Appl., 14.2.30.

Bleaching clay containing oil is heated under pressure with dilute acid to a temperature above the normal boiling point of the acid. The oil separates in a layer which can be run off. If necessary, the clay may first be extracted with a lighter solvent. T. A. S.

679. Continuous Method for Sweetening Petroleum. H. H. Cannon and W. W. Gary. U.S.P. 1,789,167, 13.1.31. Appl., 21.1.29.

A continuous process for sweetening sour oil is described. A stream of sour oil is brought into intimate contact with an incoherent comminuted solid reagent comprising lead monoxide, a hydroxide of an alkali earth metal of Group II, a light porous inert material and sulphur. The latter is continuously introduced into the zone of contact in a quantity sufficient only to sweeten the said sour oil. The flow stream of sweetened oil, free from reagent, is continuously withdrawn from the zone of contact. W. D. S.

680. Sweetening Sulphurous Petroleums. H. H. Cannon and W. W. Gary. U.S.P. 1,789,168, 13.1.31. Appl., 21.1.29.

A method of sweetening oils containing mercaptans is described. The oils are treated with a powdered solid comprising calcium hydroxide or other alkaline metal hydroxide and lead monoxide to convert the mercaptans into lead mercaptides. The lead is precipitated from the mercaptides by the addition of sulphur to the oil. Advantages claimed for the substitution of lime for caustic soda are lower cost and efficiency of regeneration of the spent earth metal reagent. Moreover, the alkali earth metal salts are inert to action on the constituents of the oil. W. D. S.

681. Oil-Refining Process. I. B. Funk. U.S.P. 1,789,173, 13.1.31. Appl., 2.10.26.

The oil is distilled in a shell still and bottoms are continuously run from the still, and the vapours and steam pass to a fractionating column. This is in two sections: the lower, a fractionating column of any desired construction, and the upper section is a condensing section. The two sections are divided by a solid separating plate, fitted with bubble capped connections between the two sections. Condensate from the upper section collects on this plate and passes to a water trap where the water is separated. Part of the condensate is returned to the top of the fractionating section as reflux, and the remainder passes through a seal and into the vapour pipe from the top of the condenser section, and together with uncondensed vapours from this section passes through water condensers to the distillate receivers. Reflux collecting in the bottom of the fractionating section is continuously withdrawn and recirculated through the still. W. L.

682. Apparatus for Dephlegmating Vapours. W. M. Cross, assr. Gasoline Products Co. U.S.P. 1,789,670, 20.1.31. Appl., 29.10.23.

The dephlegmating column consists of three sections. The middle section is composed of a number of plates. These are perforated and are fitted with short vertical risers which furnish communication between the compartments between the plates and permit the vapours to rise from any compartment to the one above. These risers are capped by cap shaped cups with serrated lower edges.

A liquid level is maintained on each plate by vertical down comers, the lower ends of which are sealed in the liquid on the plate below.

On leaving the uppermost plate the vapours are brought into contact with coils through which cooling mediums are passed.

The bottom portion of the column is a reboiling section. Reflux from the tower collects here, and is maintained at a constant level, and is heated by a reboiling header, through which highly heated oil or any suitable medium is passed.

The factors which control efficient operation are the velocity of the vapours through the column, and the definite and limited relation between the area of the surface of the pools through which the vapours pass and the size of the risers through which the vapours pass from one plate to the next. W. L.

683. Treating Oil. W. Yantis. U.S.P. 1,791,695, 10.2.31. Appl., 24.2.28.

The apparatus consists of three covered tanks of increasing capacity. A predetermined level of water is maintained in the first tank. Oil and steam are admitted to this tank below the water level through separate pipes, each of which is fitted with radially extending arms. The oil passes out through nozzles arranged horizontally and directed circumferentially to the tank, while the steam is passed through distributing heads so that it spreads in a horizontal direction. A sludge and water outlet is provided at the lower portion of this tank.

Above the water level is an overflow pipe. Oil passes through this into a vertical pipe leading to near the bottom of the second tank. The upper end of the vertical pipe supports a container, from which treatalite or soda-ash may be admitted. This tank is provided at the bottom with an outlet valve, connected to a swing pipe, whose position can be controlled by means of a windlass on the top of the tank.

The third tank is similar in construction to the second and may be used for auxiliary settling or storage. W. L.

684. Purifying Cracked Petroleum Vapours. C. R. Watson and R. C. Osterstrom, Assrs. to Pure Oil Co. U.S.P. 1,793,885, 24.2.31. Appl., 21.8.28.

Petroleum oils are passed through a heating zone where they are vaporised, and the vapours heated to above 1000° C. On leaving the heating zone, the oils are cooled to a temperature not in excess of 600° F., and the vapours remaining at this temperature passed into a fractionating zone. A quantity of refining agent (*e.g.*, fuller's earth) is introduced into the vapours between the cooler and the fractionating zone. W. H. T.

See also Abstract Nos. 627, 632.

Cracking.

685. Pyrolysis and Destructive Hydrogenation of Heavy Crude Oil Fractions. V. N. Ipatiev. *Nat. Petr. News*, 1931, **23** (12), 49; (13), 61.

Cracking and hydrogenation processes are reviewed from point of view of increasing the gasoline yield of crude. Mechanism of cracking processes can be explained simply as follows: when hydrocarbons of high molecular weight are subjected to heat and pressure, molecular rearrangement produces less stable forms which are subject to disruption, isomeric transformation of the new bodies yielding gasoline—*e.g.*, $RCH_2—CH_2—CH_2—CH_2—CH_2R = RCH_3 + CH_2=CHCH_2CH_2R$.

In practice concurrent formation of coke and gas obtains and the above ideal case is never realised. Further decomposition of the products of disruption yields bodies of still lower molecular weight. Parallel with the disruption processes dehydrogenation produces poly-aromatic hydrocarbons, which as a rule have high molecular weights and tend to run to coke. Many other aspects of the formation of coke are cited. It is claimed that from the point of view of gas formation cracking under pressure shows advantages over vapour-phase cracking. The yield of saturated hydrocarbons is increased by cyclization of unsaturateds. No difference has been observed by cracking in the presence of the usual catalysts. Details of investigation designed to determine mechanism of hydrogenation using pure materials discussed. Average minimum pressure for hydrogenation is 70 atm. Products of hydrogenation dependent on surface area of stock exposed to hydrogen. Mechanism of hydrogenation of petroleum hydrocarbons differs from that of aromatics. In the former hydrogenation occurs to form saturated bodies from unsaturated products of disruption with consequent lowering of molecular weight. Aromatics tend to form saturated hydrogenated bodies which disrupt to form lower homologues of original. The behaviour of many pure substances during hydrogenation is discussed. H. G.

686. Cracking Rumanian Gas Oil. C. H. Alberding and W. A. Keightley. Mon. Petr. Roumain, 1931, **31**, 291–292.

The article deals with the advantages of using, when the cost permits, gas oil instead of residue from petroleum in a Dubbs cracking plant. From a gas oil—S.G. 0·860—60 to 65% of petrol of high anti-knock value was obtained. The plant differs only in dimensions from the usual Dubbs installation—it has a normal capacity of 300 brls. a day. A pressure of 16 kg./sq.cm. is used.

From the gas oil, 72·5% of pressure distillate boiling from 37° C. to 245° C. (compared with 55% from the residue) was obtained, while only 15% of coke was formed (compared with 30%). After acid and alkali treatment, the pressure distillate is rectified and the residue may be returned to the cracking plant. The petrol S.G. 0·730—0·740 boils between 30° C. and 205° C.

The capacity of the plant is less per 24 hours with gas oil than with petroleum residue "on stream," but there are fewer interruptions in the former case as the cycle occupies 120 hours (compared with 48 hours for the residue), the charge being 200 tons with gas oil or 100 tons of residue.

The plant, originally designed for cracking low temperature tar, is very flexible, and has also been used for various crude oils, residues, etc. From a tar oil from Salerni 40% of pressure distillate was obtained, the petrol from this being roughly equivalent to benzene in anti-knock value.

C. C.

687. Cracking Petroleum Oils. N. V. de Bataafsche, Petr. Mij. Assees of W. J. Perolis. E.P. 343,286, 19.2.31. Appl., 23.1.30. Conv. (U.S.A.), 14.2.29.

Increased yield in cracking is obtained by carrying out the operation in two stages. In the first the oil is heated up to a temperature at which coking would take place but for the fact that the oil is highly turbulent and the heating only carried on for a short period. The oil is then passed to an evaporator, the asphalt-containing portion being removed. The heavy fractions from a dephlegmator are returned for recracking in the second stage being passed through a series of three heating coils. T. A. S.

688. Treatment of Liquid Hydrocarbons. S. J. M. Auld, A. E. Dunstan and P. H. Herrings, Assrs. to Anglo-Persian Oil Co. U.S.P. 1,798,054 24.3.31. Appl., 11.2.24.

The patent deals with a pressure cracking process for liquid hydrocarbons of the single pass type carried out substantially in the liquid phase. The liquid is preheated in a single stream to below a determined limit of the eventual temperature of cracking. The liquid is then passed in narrow streams at low velocity in a relatively short path to a reactor. The moving streams of liquid (having a very low cross-sectional temperature gradient) are subjected to heat, uniformly and gradually applied to a cracking temperature, without causing the consummation of the cracking reaction. The heated liquid is maintained in the reactor in order to bring about cracking, after which it is withdrawn. W. H. T.

689. Treating Petroleum Oils. C. B. Watson, Assr. to Gyro Process Co. U.S.P. 1,797,305, 24.3.31. Appl., 21.8.28.

Claims are made for a method of treating petroleum from which the gasoline has been removed. The oil is vaporised and then conducted to a cracking apparatus where the vapour is subjected to a temperature sufficient to break down the high-boiling compounds. The highly-heated cracked vapours are passed into a receptacle which is fitted with a closed coil. Through the latter is passed the charging stock containing free gasoline, whereby low-boiling compounds are vaporised and removed. The higher-boiling compounds are discharged into the receptacle in liquid form for admixture with the vapours discharged from the cracking apparatus. By this means the temperature of the cracked vapours is reduced and further cracking reactions are arrested. W. H. T.

Hydrogenation.

690. Hydrogenation of Petroleum. R. T. Haslam and R. P. Russell. *Petr. Times*, 1931, **25**, 371–372, 429–430.

Hydrogenation of petroleum considered under five headings: (1) Conversion of heavy, high sulphur, asphaltic crudes or refinery residues into gasolines or distillates low in sulphur, free from asphalt without concurrent coking; (2) conversion to premier quality of low grade lubricating oils; (3) improvement as regards colour and burning properties of burning oils; (4) desulphurisation, colour and gum stabilisation of low grade gasolines; (5) conversion of paraffin base gas oils into good quality gasolines.

Charging stock and hydrogen are preheated and delivered at 3000 lb./sq. in. to reaction vessel containing catalyst and thence through heat exchangers to separators. Sulphur in charging stock appears as H_2S in unconsumed hydrogen. Yields in processes 1, 2 and 3 usually above 100% volume. Hydrogenation claimed to be an improvement over present cracking process from point of view of absence of coke and waste gaseous products, larger scope, greater gasoline yield.

For lubricating oils improved temperature—viscosity relationship, Conradson carbon content, flash point and gravity claimed. Typical analyses before and after hydrogenation are given. H. G.

691. Recovery of Hydrogen from Gas Mixtures Containing Hydrocarbons. Gesellschaft für Linde's Eismaschinen A.-G. E.P. 317,731, 19.2.31. Appl., 19.8.29.

The hydrocarbons containing hydrogen and other constituents are first concentrated, and any contact poisons removed by cooling to a low temperature. They are then mixed with oxygen before passing over a catalyst for the purpose of supplying part or all of the heat required in the decomposition reaction; the gas mixture is thus decomposed catalytically with water vapour into a hydrogen and carbon dioxide. The gas leaving the contact apparatus and still containing a certain amount of unconverted hydrocarbons is again submitted to low temperature separating, concentrating and simultaneously separating hydrogen. The oxygen is obtained as a byproduct in the preparation of the liquid nitrogen used in the cooling stage.

W. S. E. C.

See also Abstract No. 685.

Chemistry of Petroleum.

692. Kinetics of the Thermal Decomposition of Methane. T. S. Wheeler. *Fuel*, 1931, **10**, 175–181.

A critical examination is made of the results given in the literature on the thermal decomposition of methane with a view to obtaining some information as to the kinetics of the reaction. The latter part of the reaction is first considered, and whereas Bone and Coward, and also Wheeler and Wood, indicate a reaction of zero order, Holliday and Exell make no mention of this. From the same workers' experiments, equations are deduced for the initial rapid decomposition (which is retarded by the hydrogen produced), which satisfactorily represent the available kinetic data on the decomposition of methane although the type of equation is difficult to explain by simple physical mechanism.

In contact with silica, porcelain and chrome iron the reaction is heterogeneous, and apparently methane decomposes to hydrogen and the radicals $-CH_2$, $=CH_2$, $\equiv CH$, which are unstable, forming carbon and hydrogen or uniting to form ethylene and acetylene from which aromatic hydrocarbons may be produced. As hydrogen displaces methane from the surface, the chance of two radicals meeting decreases and they decompose directly to carbon and hydrogen. When the surface becomes completely covered with hydrogen, a slow zero order reaction is usually observed, presumably due to absorption of methane on parts of the surface which do not absorb hydrogen. Further lines of investigation are indicated.

C. C.

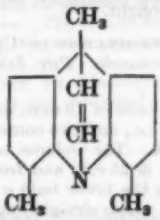
693. Nitrogen Compounds in Petroleum Distillates. III. Structure of a Hydroaromatic Base of the Formula, $C_{16}H_{23}N$. W. C. Thompson and J. R. Bailey. *J. Amer. Chem. Soc.*, 1931, **53**, 1002–1011.

Further attempts have been made to determine the structural formula of a hydroaromatic base of the formula, $C_{16}H_{23}N$, obtained from a petroleum distillate (*J.A.C.S.*, 1930, **52**, 1239). The base shows unusual stability towards permanganate except in acid solution. It is very resistant to oxidation with chromic acid-sulphuric acid solution. Nitric acid at 170° C. gave four nitration products though the yields were poor. The final oxidation product with nitric acid was pyridene-2·4·5-tricarboxylic acid, *i.e.*, berberonic acid, indicating that the nitrogen in the $C_{16}H_{23}N$ base is in a six-membered ring. The location of a methyl group at the *p*-position in the piperidene ring harmonised with the failure to isolate a trimethyl-trinitro derivative among the nitric acid reaction products.

All hydrogenation and dehydrogenation experiments failed to effect a chemical change in the substance. Products such as heptamethyltetrahydroquinoline or a trimethyldecahydroacridine were therefore improbable. The chemical behaviour of the $C_{18}H_{31}N$ compound, where its basic properties were not involved, strongly paralleled methylcyclopentane. Thus a condensation of two cyclopentane nuclei with a piperidine nucleus was tentatively assigned. Such a formula harmonises with the nitration products.

The only unsaturated group is assumed to be an etheno bridging between the nitrogen and the *p*-carbon in the piperidine ring. Steric hindrance is suggested to account for instability to oxidising agents.

Thus the base is regarded as 4-methyl-14-etheno piperidine condensed at 2, 3, as well as at 5, 6, with methylcyclopentane. In addition to the two cyclopentane nuclei, this structure represents two tetrahydro pyridine compounds within the piperidine periphery, so that in all five rings are involved. The following is the structural formula assigned :—



In order to obtain a simplification of the nomenclature of the products obtained, the compound $C_{18}H_{31}N$ is supposed to be based on the parent compound *pyrindacine*, so that the name decahydro- 3,5,8 trimethyl-4, 8-ethenopyrindacine is applied to the above structural formula for $C_{18}H_{31}N$. *Pyrindine* is proposed as a name for a single cyclopentane ring attached to a pyridene ring. W. D. S.

694. Formation of Peroxides by the Direct Oxidation of Hydrocarbons by Air. P. Mondain-Monval and B. Quanquin. *Ann. des. Comb. Liquides.* 1930, **5**, 915–917.

When pentane, hexane, etc., or gasoline vapour mixed with air is passed through a tube at 300° C., besides a rise of temperature, white fumes, a bluish luminescence and the formation of aldehydes and CO_2, the condensation of yellowish oily drops is observed. This oily substance is slightly acid and is soluble in water and alcohol, but not in hydrocarbons. It contains peroxides, liberating iodine from potassium iodide, giving a blue colour with dichromate and ether, and oxidising titanous chloride and hydroquinone.

With alkaline hydroxides a vigorous reaction takes place, resulting in the evolution of hydrogen and the formation of sodium formate and methyl alcohol. On heating, the oily substance decomposes exothermally, giving a blue luminescence, white fumes and aldehydes. The preparation was then carried out so as to keep the temperature of the tube a few degrees below that required to produce white fumes. In this way 70 g. of hexane yielded 20 to 25 c.c. of oil. The oil also decomposes on standing, yielding aldehydes.

It is considered that the peroxides present belong to the series R.O.O.H., the monomethyl and ethyl members at least being present. The properties such as formation of formate, methyl alcohol and hydrogen with alkalies, are similar to those of the compound $CH_3.O.OH$, studied by Rieche and Hitz (Ber., 1929, **62**, 2460). The formation of these peroxides has a bearing on the phenomena of detonation in internal-combustion engines. E. B. E.

Analysis and Testing.

695. Copper Tarring Number. K. Typke. *Petr. Zeit.*, 1931, **27**; Motorenbetrieb (2) 3–7.

The differences in the results obtained by the sheet-copper and Kissling's method are discussed, and also the essential factors influencing the results. Various data for Russian and American oils are tabulated.

The quantity of oxygen used and time of oxidation were found to be of great importance. A gravimetric determination of the sludge formed showed an increase in value on standing. It is emphasised that three benzol washings are necessary after oxidation and standing, and after the third washing the benzol should be left for a longer time in contact with the acidified lye. Electrolytic copper was found to have a catalytic effect. It is specified that the copper used should be 10 cm. long and 0·1 mm. thick.

According to this method the tar value for a good oil should be below 0·4 and sludge below 0·1% by weight. W. S. E. C.

696. Burette for the Determination of Unsaturated and Aromatic Hydrocarbons. Dr. Formanek. *Petr. Zeit.*, 1931, **27**; Motorenbetrieb (2), 7.

The apparatus consists of a tube of 10 mm. internal diameter which reads from 0 to 15 c.c. in tenths of a c.c., and two consecutive bulbs *a* and *b*; below the lower bulb is a stop-cock. The volume capacity measured from the upper to the lower zero mark is 25 c.c., and from the bulb *a* to the zero is 25 c.c. In the actual analysis the lower bulb *a* is filled with sulphuric acid at 20° C. by means of a funnel, whose straight end reaches the lower mark and then with benzine also at 20° C. The acid and benzine are mixed intimately, and the readings taken when the burette has been cooled to 20° C.

The apparatus has been used for various benzines and benzine-benzol mixtures. W. S. E. C.

697. Luminiscence of the Vapours of Some Cyclic Hydrocarbons in the Electrodeless Discharge. Aubert & Vencov. *Ann. des. Comb. Liquides*, 1930, **5**, 781–794.

It has been observed by several authors that gases become luminous when the glass tube containing them is placed in a high frequency alternating field. The object of this work is to use the effect to determine the constituents of hydrocarbon mixtures.

An apparatus is described which allows the examination of hydrocarbons in an alternating field of frequency about 10^4, attention being paid to the emission spectra caused by Tesla excitation and the absorption spectra both of liquid and vapour.

With benzene it was found that the maximum luminosity was produced at 0·5 to 1·6 mm. pressure, the appearance being bluish-white. The spectra showed a number of narrow bands which were arranged in seven groups between about 3121 and 2667A. The number of emission bands of benzene is not well known, but from 52 to 450 can be counted in intense fields.

For observing the absorption spectra, light from a hydrogen lamp was employed, and 5 or 6 characteristic bands were noted. The positions varied slightly according to whether a solvent (methyl or ethyl alcohol, etc.) or no solvent was employed. Similar effects were obtained for the vapour.

Cyclohexane showed no effects except those due to traces of benzene present as impurity. Cyclohexene was also examined, and the results are given in tables and in photographs of the spectra. The method is very sensitive for the detection of traces of benzene. E. B. E.

698. Necessity of Employing the Co-efficient of Absolute Viscosity or the Coefficient of Absolute Kinematic Viscosity for the Practical Measurement of Viscosity. P. Woog, *Ann. des. Comb. Liquides*, 1930, **5**, 805–829.

After discussing the importance of viscosity in technical work, the author proceeds to criticise commercial instruments on the following grounds: the effect of temperature on the viscosity of oils is very great, but the temperature regulation of commercial viscometers is usually not good; the jet of oil emerging from the instrument enters the air, instead of the interior of the liquid as it should, and hence capillary forces come into play to an extent depending on the speed of outflow; the shortness of the tube and the relatively high speed of flow cause errors due to turbulence.

The various important commercial instruments, including the Barbey Ixometre, are described, and then the laws of Poiseuille and Stokes are treated.

The use of calibrated Ostwald viscometers is recommended as being quicker, easier to use and more accurate. The results should be expressed in "poises" or "stokes," the latter being the name proposed by Brillouin for the unit of absolute kinematic viscosity.

Extensive conversion tables for kinematic viscosity, Engler, Barbey, Saybolt (Universal and Furol) and Redwood (No. 1 and Admiralty) results are given. E. B. E.

Anti-Detonation.

699. Anti-knock Fuels and Engine Design. E. Bartholomew and F. Fricker. *Oil & Gas J.*, 1931, **29** (45), 38.

The automobile and aircraft industries are faced with the same fundamental problems—viz., economy in the production of power, in respect of fuel, space and metal. For automobiles this is expressed in the form of a demand for greater speed, accelerating power and economy, whilst commercial aviation requires larger pay loads which can only be obtained by lightening the plane or diminishing the weight of the power plant per h.p. developed. Co-operative research now being undertaken by the oil and automotive industries will greatly assist the efficient utilisation of scientific developments in both industries.

The beneficial effect of increasing the compression of Otto cycle engines on efficiency and specific fuel consumption is considered from the theoretical standpoint and related to actual practice. Comparison is made with Diesel engine efficiencies showing that Otto cycle engines having a compression ratio of 8/1 are as efficient as Diesel engines having a compression ratio twice as high, and possess other advantages for light weight automotive vehicles. Since the anti-knock value of available fuels has increased of recent years, and will almost certainly tend to increase further, the building of engines having higher c.rs. than hitherto is possible. Some of the problems which arise are discussed. Intake manifold temperatures now attained in certain engines cause pre-ignition when compression pressures are increased, regardless of the anti-knock value of the fuel used. The important effects observed in the case of an 8-cyl. engine used for test purposes are shown, and suggest the desirability for an investigation of the possibilities of cold carburation in connexion with supercompression engines. Other factors which assume greater importance are: carbon formation and removal, the maintenance of close

limits on combustion chamber volume, ignition timing, cylinder size and rate of change of pressure rise. The influence of each factor is mentioned.

Charts are presented to show the variations in the heat balance and the h.p. developed at 1000/3000 r.p.m. by an 8-cyl. engine at c.rs. of 5·2, 6 and 7/1. Increase in the ratio is accompanied by increase in h.p. and decreases in exhaust temperatures, fuel consumption per h.p. hour, and the amount of heat supplied to cooling water. Since the friction losses in the chassis at low speeds represent a large proportion of the power developed, the increase in power output due to the increased compression ratio constitutes a large percentage of the power available for acceleration. A chart shows the increased accelerating ability of the 8-cyl. engine; up to 45 m.p.h. there is a gain of 20% by increasing the ratio from 5·2 to 7/1. This can be obtained without increase in cost of the engine, so that the cost per h.p. and per unit of acceleration is decreased.

Detonation and overheating due to the use of improper fuel is doubtless the cause of a large number of airplane disasters. The difficulty has been that neither the engine builder, airplane operator nor fuel vendor has been able adequately to express the anti-knock value of the fuel required or offered, since no universal standard was available. A common basis of expression is therefore essential. Aircraft engines operating at high temperatures introduce a fuel problem on account of the danger of high-cylinder temperatures causing piston seizure. Benzol blends cause much higher cylinder temperatures than ethyl gasolines having similar anti-knock value when tested in equipment designed for evaluating fuels under automobile operating conditions. In addition, the increase in anti-knock value obtained by the use of benzol diminishes in comparison with that obtained by the use of L.T.E. as the cylinder temperature increases. It appears possible that tests of anti-knock value at two widely different jacket temperatures may be necessary.

The methods which can be utilised for increasing the anti-knock value of fuels are reviewed, and the tendency for certain cracked gasolines to depreciate in anti-knock value and to form gum on storage is pointed out. The basis of evaluation should be on the fuel as it reaches the customer and not on the freshly produced refinery material. R. A. E.

700. Knock in Internal Combustion Engines and the Action of Anti-knock Compounds. J. Lorentzen. *Zeit. Angew. Chem.*, 1931, **44**, 130–136.

Experiments on the combustion of mixtures of hexane and pentane with air in various proportions, and study of the time-pressure curves obtained, indicate that when knock occurs reactions take place which differ from the normal. Variation of the position of the ignition spark shows that knock is produced only when a wave of incomplete combustion travels through the cylinder, and is the result of the explosion of the intermediate products of combustion by a reflected compression wave when it meets the combustion wave. The normal velocity of propagation of the flame is approximately doubled when knock occurs. The production of turbulence in the gas mixture prior to ignition increases the speed of combustion, and under these conditions knock occurs only at initial pressures greater than 8 atm. If lead tetraethyl be mixed with the fuel before it enters the cylinder knock is not inhibited, but by introducing the products of combustion of the lead compounds into the cylinder before ignition of the mixture knock is inhibited at pressures below 5 atm., whilst if turbulence is set up in the mixture knock does not occur at pressures below 9 atm. The theory of the action of anti-knock compounds is discussed. W. S. E. C.

701. Molecular Structure and Knocking. T. A. Boyd, *Oil and Gas J.*, 1931, **29** (42), 147.

Molecular structure has a great influence on the anti-knock value of a hydrocarbon or compound, as is clearly demonstrated by the difference in the anti-knock values of ethyl ether and butyl alcohol, which have the same chemical composition. A study of the subject has been made, and it has been shown that the supposition that all paraffin hydrocarbons possess low anti-knock value is incorrect. Three general rules have been evolved as a result of the research: (1) Straight chain paraffin hydrocarbons show a regular and rapid decrease in anti-knock value with increase of mol. wt.; (2) comparing paraffin hydrocarbons of equal mol. wt., the introduction of methyl groups gives an increase in anti-knock value; (3) comparing paraffin hydrocarbons of equal mol. wt., anti-knock value is improved as "centralisation of the molecule" is increased. Thus there are nine possible forms of the hydrocarbon C_7H_{16}, of which *n*-heptane has the lowest, and the form in which the seven carbon atoms are most closely centralised, the highest anti-knock value.

n-Heptane can only be used efficiently in an engine having a compression ratio not higher than 3/1, but the most molecularly centralised hydrocarbon could be used in engines having a compression ratio of 12/1 although this is a higher ratio than has ever been employed for automobile engines. The results suggest that Pennsylvanian gasolines are not composed to a large extent of straight chain paraffin hydrocarbons. R. A. E.

702. New Developments in Knock Rating. G. Edgar. *Oil and Gas J.*, 1931, **29** (42), 101.

Comparison of the anti-knock value of two fuels in a single cylinder knock testing machine may be effected in many ways, but fall into two main classes:—

(*a*) Determining the variation in some single engine factor such as throttle opening, compression ratio, spark advance, speed or load, necessary to make the two fuels knock equally.

(*b*) By comparing the anti-knock value of each fuel under fixed engine conditions, with those of a series of fuel standards of known anti-knock value. The results are expressed in terms of the mixture of standard fuels which equals the gasoline in anti-knock value.

Different laboratories have used numerous different standards in applying method (*b*), and this has resulted in confusion since the values reported by any particular laboratory cannot be assessed by anyone who is not acquainted with the method employed. Some primary standards of anti-knock value, reproducible at any time, are therefore essential for the sake of uniformity of expression of results. In 1926 the author proposed the use of normal heptane and iso-octane, respectively lower and higher in anti-knock value than any commercial gasoline, for this purpose. The use of these standards has now been recommended by the C.F.R. Steering Committee, which has also recommended that all knock ratings should be expressed in "octane numbers," defined as "the percentage by volume of *iso*-octane in blends of this substance with normal heptane necessary to equal the anti-knock value of the fuel in question." This has also been approved by the S.A.E.

Provided the engine conditions are kept absolutely constant when matching the fuel against iso-octane/heptane blend, the octane numbers obtained by almost any well-recognised method of measurement will agree reasonably well.

Since the pure hydrocarbons are very expensive, suitable calibrated secondary standards are desirable for routine testing. These can be prepared from selected stable gasolines of appropriate anti-knock value, but it is

desirable that all laboratories should use the same secondary standards as far as possible. The Standard Oil Development Co. have prepared 30,000 gal. of two such standard reference fuels, having octane numbers in a particular engine of 50 and 68 respectively, which can be purchased by anyone interested. Curves showing the octane number rating of blends of these two fuels, and of blends of each with L.T.E. and benzol are given. It is probable that other secondary standards of general availability will shortly appear. R. A. E.

See also Abstract No. 630.

Engines.

703. Fuel Injection with Spark Ignition in an Otto-Cycle Engine. C. F. Taylor, E. S. Taylor and G. L. Williams. *J. Soc. Aut. Eng.* 1931, **28**, 345–350.

An N.A.C.A. universal test engine has been adapted for use with fuel injection in place of the normal carburettor. A special injection valve was developed, spring-loaded to open at a pressure of 3000 lbs. sq. in., the fuel being supplied by a Bosch fuel-pump operated from an engine camshaft through a gear-coupling to permit adjustment of the injection timing. The valve showed good atomisation characteristics with low penetration.

The first tests were carried out with gasoline injected into the inlet-pipe, during the suction stroke, beginning 30° after top-centre and continuing for about 21°. A gain of about 6·65 per cent. in volumetric efficiency and 6.5 per cent. in maximum B.H.P. is obtained and is considered to be due to the removal of the carburettor. The amount of fuel injected per stroke is probably also much more nearly constant.

Injection was then tried with the valve in one of the spark-plug holes at the top of the cylinder. A still further increase in volumetric efficiency was observed, this being due to drop in temperature and pressure due to cooling by the injected charge; this drop in pressure is shown mathematically to be about 16 per cent., or 2 lb./sq. in. The performance is much better than with carburation, maximum power being about 11 per cent. greater, and could be improved by introducing good turbulence. Knocking characteristics are not affected whether the fuel be introduced by carburation or injection.

It was found possible to run on fuel-oil in the same engine with injection into the cylinder, the compression ratio being reduced to 3.5 : 1 to avoid detonation. The volumetric efficiency is very much less than with gasoline. The engine could be started from cold on fuel-oil, but some trouble was experienced from fouling of the plug-points during the warming-up period. E. B. E.

704. Note on the Power Recoverable in the Exhaust Gases of Two-stroke Diesel Engines. Guillet. *Rev. Petrolifers*, 1931, (414), 300.

In a study of the above subject, a theoretical determination of the maximum power that can be obtained in using the exhaust gases in a variable pressure turbine appears to be of value.

For an engine of compression ratio 12 to 1, the efficiency of the Diese cycle is calculated to be 0.55, while that of the explosion cycle is 0.64—the gain in efficiency by recovering the lost power is about 17 per cent. For a 2-stroke engine, there is a gain of about 24 per cent.

It is calculated on certain assumptions that 0.33 h.p. per effective h.p. can be recovered in using all the available energy in the exhaust gases, but allowing for various losses this becomes 0.176 h.p.

Further, a turbo compressor of efficiency only 0.36 would suffice to produce the power necessary to compress the air required, whereas the efficiency of the turbo compressor suggested by Rateau for supercharging a 4-stroke Diesel exceeded 0.60.

It is possible, therefore, with a 2-stroke engine to carry out scavenging and supercharging by recovering a part of the energy in the exhaust gases. C. C.

705. Influence of the Piston on the Consumption of Oil. Dintilhac. *Rev. Petrolifere*, 1931, (415), 331. (From a lecture at the Société des Ingenieurs de l'Automobile.)

Any oil which passes between the piston and the cylinder walls into the combustion chamber is lost. To reduce this to a minimum, two suggestions are made: (1) Limit the oil entering by chamfering the base of the skirt of the piston; (2) arrange drainage holes inside the piston to return the excess oil to the sump. This may be done by arranging lateral ducts under the piston rings. The grooves of the first and second rings should not be pierced or tightness may be sacrificed and dilution of the oil may follow. Alternatively, grooves may be traced on the cylinder and pierced with holes at intervals to remove the oil which collects.

Worn piston rings may allow oil to pass, while if the rings are slack in their grooves the motion of the piston causes the rings to act as a pump. M. Dintilhac tested his theories by running a car on the track. With an aluminium piston there was a minimum consumption at 40 km./hr., a fact accounted for by the expansion of the metal. It was also shown that the grooves and holes in the piston diminished, and made more regular, the consumption of oil. C. C.

706. Friedl Exhaust Valve Cooling System. T. Lee. *Aviation*, 1931, **30**, 186–187.

This system provides air cooling of exhaust valves. The cooling air enters holes in the tappet end of the valve stem and passes through the hollow centre of the stem, and leaves through holes under the head of the valve, to mix with the exhaust products of the cylinder. The cross sectional area of the entry holes, the hollow passage in the stem and the exit holes, progressively increases to allow for expansion of the air as it travels from the cooler to the hotter parts. A positive flow of cool air from the tappet end to the head end is accomplished by increasing the normal pressure in the rocker box by means of a small air scoop at the top end of the box, and by lowering the pressure in the exhaust port by mounting individual venturi exhaust stacks on each cylinder exhaust port.

At flying speeds of 100 m.p.h. an average vacuum of 12 in. of water is maintained in the exhaust passage. There have been no indications of (1) exhaust products having entered the valve stem during the early interval of exhaust valve opening; (2) valve warpage which would result if cold air impinged on the hot valve head. During flight tests on a Boling 50-B mail 'plane (Pratt and Whitney Wasp engine) full throttle engine speed on ground was increased by 90 r.p.m. and in the air by 125 r.p.m. as a result of the replacement of the standard valve equipment by valves of the above design. 'Plane speed at full throttle was also increased by 3 m.p.h.; engine acceleration was quicker and more positive, and a saving in weight of 56½ lbs. was effected. C. I. K.

707. De La Vergne and MWM-Bene High Speed Diesel Engines. *Mech. Eng.*, 1931, **53**, 220; *Diesel Power*, 1930, **8**, 622–624, 626–628.

The De La Vergne engine is a 6-cyl. American unit with a 9 in. bore and 11 in. stroke, and is rated at 300 b.h.p. at 750 r.p.m. It is designed with airless-injection combustion system.

The shape of the combustion-chamber is defined by a flat-top cylinder head and a conical, slightly shrouded piston, whose contour concentrates the combustion air in the paths of the several jets. The construction permits the clearance needed for a compression ratio of 13 : 1 without resorting to an excessively flat combustion chamber.

The spray valve is of the differential needle type, and is timed and operated without valve gear by means of an individual Bosch pump, each cylinder having mounted in front one of these pumps, which are described in detail in the original article.

Four valves are fitted in the cylinder head located in such positions that the cored intake passages in the cylinder heads leading up to them cause rotation of the air-charge in the cylinder.

The ratio of cylinder centre distance to cylinder bore is 14·5 : 9 or 1·6. Low ratios of this order are considered a growing feature of light-weight high speed engines, as stiffness is imparted to the crankshaft and reduction obtained in weight and length. The rates of fuel consumption are claimed to correspond to 0·40, 0·41 and 0·44 lb. per b.h.p.-hr. at full, three-quarters and one-half load.

The MWM-Benz is a high speed automotive type engine with pre-compression chamber, primarily developed for use on fast auxiliary boats.

It is a four-stroke cycle vertical engine with 6 or 8 cylinders of 8·07 in. bore, 10·63 in. stroke running at 800–1000 r.p.m., developing 250 b.h.p. in the 6 cyl. unit at the lower speed and 300 at the higher speed with a weight of 6170 lb.

Each cylinder has its own cylinder head, inside of which is located a pre-combustion chamber with fuel valve and atomizing thimble as well as inlet and exhaust valves.

The pre-combustion chamber is eccentrically located with respect to cylinder centre line; it is therefore possible to keep the valves so large that good volumetric efficiency is assured.

Fuel pumps for all cylinders are united in a common block fixed to the front end of the engine; these are driven from the camshaft by means of helical gears.

Fuel consumption is claimed to be about 0·436 lb. per b.h.p.-hr.

W. A. W.

708. Two-stroke Cycle Internal-combustion Engines. S. Miller. E.P. 340,745, 8.1.31. Appl., 12.12.29.

This invention, which is an improvement on a prior specification (10,998 of 1908) relates to the use of a scavenge pipe, preferably of curved section, mounted near the periphery of the piston of a 2-stroke engine. This pipe, which continues through the piston wall, communicates with the scavenge port as the piston nears its outer dead-centre position. In this manner the scavenge air is directed towards the cylinder head and away from the exhaust ports in a more efficient manner than by the usual baffle. G. B. M.

709. Ports and Passages of Cylinders of Two-stroke-cycle Internal-combustion Engines. Sulzer Frères. E.P. 341,782, 22.1.31. Appl., 21.3.30.

Better scavenging and less waste of scavenge air in a 2-stroke engine is claimed for this invention, which comprises a combination of a V bend in the air-supply pipe with an additional bend just before it joins the scavenge port. In this manner a zig-zag or N-shaped conduit is formed, of which the final branch is inclined obliquely towards the cylinder head. In addition the lip of the piston at its outer dead centre position partially masks the scavenge port and so gives a venturi effect. G. B. M.

710. Filters for Petrol. A. G. Fenn and Anglo-American Oil Co. E.P. 343,219, 19.2.31. Appl., 5.12.29.

The filter comprises an outer case formed in two parts and provided with means for their ready connection or disconnection, together with admission and discharge openings. Perforated conical bodies are situated within the case, and are so positioned as to cause the fuel to pass through them. Filtering material is packed both inside and outside the conical bodies, and the components of the case are held together by means of a flexible band provided with means for tightening. W. H. T.

See also Abstract No. 699.

General.

711. Useful Horizontal Tank Chart. W. F. Schaphorst. *Ind. Eng. Chem.*, 1931, **23**, 314-315.

A chart is given from which the number of gallons of liquid in any horizontal cylindrical tank can be obtained without the use of tables, etc. The limits of reading are: depth of liquid—0.1 in. to full capacity of tank, dia.—2 in. to 200 in., length of tank—10in. to 100,000 in. The chart can also be used to compute the full or partial capacity of pipes. C. C.

712. California Citrus Crops saved by Oil. L. P. Stockman. *Oil and Gas. J.*, 1931. **29** (43), 91.

The petroleum industry saved the citrus industry of California from loss by an unusually heavy frost last December by rapid supply of heater oil to all parts of the state in specially chartered trains which were given precedence on the railway lines.

The sudden, large and prolonged demand owing to the long duration of the frost made these emergency measures necessary as stocks were depleted. There are three million orchard heaters in use in California citrus groves requiring about twenty-two million gal. of oil per filling. Each heater burns from 10/15 hrs., according to the amount of heat desired, on one filling. Approximately 30% of the California citrus acreage is equipped with heaters which are lighted when the temperature drops below 28° F. so that the demand for oil fluctuates according to atmospheric temperature. A close watch on temperature conditions is maintained by the fruit growers and the U.S. Department of Agriculture. Specifications for heater oil vary, but maxima of 3% asphaltic residue and ¼% of sulphur are generally required. The oils actually supplied normally contain less than ½% carbon residue. The average price of the oil over a 15-years period is $1.30 per brl. f.o.b. refinery with minima and maxima of 50 c. and $2.08 respectively. Many types of heaters are employed, but the general type operates on an oil refined to specification in regard to calorific value and gravity (usually 27° A.P.I.).

Many of the older types of heater evolve a heavy smoke screen, which has created a nuisance in the towns, so that tests have been initiated in order to discover the relative values of smoking and non-smoking heaters as frost preventives. If the smoke screen is found to be unnecessary, it is proposed to seek legislative action to have the smoking type abolished. R. A. E.

713. Apparatus for Indicating the Presence of Inflammable Vapours or Gases. H. T. Ringrose. E.P. 344,287, 5.3.31. Appl., 9.5.30.

The patent deals with improvements in the apparatus. The walls of the porous vessel or combustion chamber being adapted to afford an unobstructed view for inspecting the interior thereof. The upper portion of the walls is of

porous pot and the lower portion of glass or other transparent non-inflammable material. The upper and lower sections are kept in alignment by a metal ring having an inwardly projecting flange, whilst rubber or other packing is used to ensure gas-tight joints. W. H. T.

714. Treating Bituminous Sands. K. A. Clark, Assr. to Univ. of Alberta. U.S.P. 1,791,797, 10.2.31. Appl., 5.5.28.

The patent describes a process of separating bitumen from bituminous sand, silt or clay in which the material is mixed with a reagent giving an alkaline reaction on hydrolysis. The mixture is introduced into a body of hot water in order to effect separation of the bitumen from the sand, etc. A water-soluble inorganic compound of a polyvalent metal is introduced into the hot water in order to coagulate the reagent and the silt or clay, the bitumen being recovered from the surface of the water. W. H. T.

Coal and Shale.

715. Researches on Low Temperature Tars. G. T. Morgan. *Fuel*, 1931, 10, 183-189.

It was found that the usual distillation methods of separation led to profound changes in the chemical nature of the components of these tars, giving also a high proportion of pitch. The method adopted, therefore, was to distil up to 120° C. to remove water and "first light oil," and then resolve the phenols and bases with aqueous alkalies and dilute mineral acids. The residual neutral oil was distilled under 2 mm. pressure up to 120° C. ("second light oil").

The material soluble in caustic soda contains true phenols and also non-phenolic substances soluble in aqueous alkali phenoxides. These non-phenols are removed by heavy organic solvents or by saturating with salt.

Among the products isolated were phenols, cresols and xylenols, together with volatile tertiary amines from the bases. From the first light oil unsaturated compounds were isolated by methyl alcoholic mercuric acetate at low temperatures, conversion to iodide and regenerating the unsaturateds by hydrochloric acid. Benzene and toluene were also identified. The second light oil contained naphthalene while from the heavy neutral oil, wax was obtained together with amorphous resinous bodies. Anthracene and some derivatives have also been identified.

The organic constituents of aqueous liquor of low temperature tars have been isolated and from a comparative study of liquors from different types of coal, it was shown that the total phenolic content was roughly proportional to the oxygen content of the coal.

A similar investigation was carried out on a primary tar and the corresponding aqueous liquor.

Comparative figures are given for the proportions of the various constituents isolated from two tars obtained from the same coal but carbonised at different temperatures. C. C.

716. Phenol Content of the Tars and Oils Derived from Coal. J. L. Wiltshire. *J.S.C.I.*, 1931, **50**, 125T-128T.

An interesting historical survey is given, which indicates that in all cases the determination is carried out on a distillate from the original tar, the total phenolic compounds being separated from hydrocarbons and bases by sodium hydroxide. Generally the phenol is separated from the total tar acids into a relatively simple mixture, and then determined by a crystallising-point method.

For mixtures containing small percentages of phenol, the method suggested is to distil the tar-acids to 230° C., and then re-distil the distillate. This was fractionated through an 8-pear column to 209° C., and the final distillate crystallised with known weights of pure phenol, sufficient phenol being added in each case to bring the total phenol content of the mixture above 80%, to eliminate the effects of varying proportions of the three cresols and the small amounts of xylenols present. A table is given showing results of three trial experiments of this method using in each case samples of the same tar acids, but two of them with known quantities of phenol added. The agreement obtained between these experiments is quite good.

The apparatus used for the determination of the crystallising-point, and the crystallising-point table, are taken from *Standard Methods of Testing Tar and Its Products*, 1929, p. 139.

The tar acids used in these experiments were prepared from tar obtained by low-temperature carbonisation of coal from the "slate" section of the Warwickshire thick-seam in semi-continuous vertical retorts (Fuel Research Technical Paper No. 17) the temperature not exceeding 625° C. The tar acids represent approximately 15% of the dry tar, the phenol content of tar being about 1%.

Details are given of the method for isolating anhydrous tar acids from any tar or hydrogenation product. W. A. W.

717. Tar Oils from the Low Temperature Distillation of Carbonaceous Materials. A. S. Ramage. E.P. 340,512, 17.12.30. Appl., 17.9.29.

Tar oil vapours from low temperature tars are contracted with metallic iron at a temperature of 1100–1200° F. in presence of water gas. After condensing and scrubbing, the aromatic hydrocarbons are separated by distillation and used as blend stocks while the residue may be used as a drying oil for varnish and paint. H. C. R.

718. Separation of Solid Constituents from Residues Obtained in Destructive Hydrogenation. I. G. Farbenind. A.-G. E.P. 341,451, 14.1.31. Appl., 14.10.29.

When carbonaceous materials are subjected to destructive hydrogenation a residue remains behind, after separation of the light fractions, which contains quantities of finely divided or even colloidal constituents which are difficult to separate.

Residues of this nature may be converted into an easily separable form by heating under pressure, until the solid constituents have coagulated to form coarse particles at temperatures higher than those employed in the destructive hydrogenation, for example, at 20° to 60° C. higher.

The mixture should not be heated to such a temperature that the whole mass is carbonised or at which substantial cracking takes place.

A refractory porous substance such as Florida earth or iron powder may be added to facilitate the separation. For example, a residue containing 38% of coal, rich in ashes, and having particles of 0·8μ cross sectional size, and which had a softening point of 90° C., is heated to 450° C. for one hour in a rotary autoclave. A pressure of about 70 atm. is set up in the autoclave. In this manner the cross section of the particles increases to 5·2 and the softening point falls to 10° C. The product may be easily filtered at 60° C., whereas before treatment it could be scarcely filtered at all.

By the addition of fine powder obtained from iron carbonyl the time of reaction may be reduced to one-fourth or the reaction temperature reduced to 20° C.

The same residue may be mixed with 2% of bog iron ore, nitrogen is forced into the autoclave up to 200 atm. pressure, and the whole is heated for 1½ hours at 450° C. The cross sectional size of the particles increases to 7·5, and the product can be centrifuged at 70° C. with recovery of pure oil. R. P.

719. Drying and Low Temperature Carbonisation. J. Y. Johnson. E.P. 341,606, 22.1.31. Appl., 8.11.29.

An apparatus is described consisting of several separate heating surfaces inclined at an angle down which the raw material moves and is heated by the counter current of hot gases flowing upwards. Provision is also made for agitation of the heating shelves. H. C. R.

720. Retorts for Low Temperature Carbonisation of Carbonaceous Materials. Carlton Main Colliery Co. E.P. 342,104, 29.1.31. Appl., 7.1.30.

A detailed description is given, together with diagrams of the apparatus, of retorts for the low temperature distillation of coal, etc. The raw material is heated in the annular space between two vertical concentric chambers and means are provided for movement of the inner one in order to discharge the carbonised product. Ribs on the surfaces of the chambers facilitate this discharge through a hinged door in the lower end of the outer chamber. The gases are passed into the inner chamber, and serve to heat the outer charge, and, in some cases, a further charge of raw material in the inner chamber itself. A water seal provides control of the volatile products. H. C. R.

721. Coking at a Low Temperature. Patent aktiebolaget Grondel-Ramen. E.P. 342,258, 26.1.31. Appl., 26.10.29. Conv. (Sweden), 25.5.29.

The coking of coal at a low temperature is described, in which coal of high content of volatiles is heated preliminary to a temperature of 250–300° C. After the desired content of volatiles is obtained the temperature is raised and coking takes place, with the formation of a solid and resistant coke. The apparatus consists of a longitudinal oven, through which the raw material is conveyed on an endless belt of special construction. Suitable inlets and outlets for heating gases enable the proper temperature gradient to be obtained. H. C. R.

722. Extraction of Hydrocarbons from Coal. F. G. Renou. E.P 342,320, 2.2.31. Appl., 1.10.29.

Pulverised coal is mixed with tar, preferably coal tar, and the whole heated to 300° F., meanwhile being mechanically agitated and circulated. The vapours are produced and drawn off by suction, and are condensed in the usual way. The residue in the retort is an efficient substitute for bitumen, and by admixture with various filling materials a suitable road surfacing material may be obtained. H. C. R.

723. Retorts for the Carbonisation of Coal. Chamber Ovens, Ltd., A. H. Lymn and N. J. Bowater. E.P. 342,720, 4.2.31. Appl., 4.11.29.

An apparatus is described in which coal and like materials are carbonised, wherein tubular heat transfer elements are built, for the purpose of cooling the charging floors and the tops of the retorts. Steam is passed through these tubular elements and the superheated vapours serve to generate water gas on introduction into the retorts. H. C. R.

724. Tar Refining. J. H. Brégi. U.S.P. 1,791,052. 3.2.31. Appl., 11.12.28.

A treatment is outlined for the refining of tars—particularly those obtained from lignite, shale and coal. The preliminary treatment is a non-destructive distillation, using superheated steam at first and then vacuum for the lubricating oil fractions. Care is taken to ensure that no resinous matter distills over into the lubricating oil fraction which is given a mild acid treatment first to remove organic bases and then a strong alkali treatment to remove the creosote oil The wax is then removed by dilution, chilling, and pressing, or in the case of amorphous wax by centrifuging. Refined oils are obtained from the dewaxed oil by acid and clay treatments, while the usual byproducts are also obtained. Variations in the above scheme include the removal of the acids and bases after dewaxing, and also the division of the lubricating oil fraction into parts by the primary distillation, and subsequent separate treatments of these parts. It is claimed that the removal of the organic acids and bases facilitate the dewaxing process and that yields are obtained comparable to those obtained from good average petroleum. H. C. R.

725. Extraction of Oils from Solid Residues obtained in the Destructive Hydrogenation of Coal. K. Gordon, H. Harper, W. I. Jones, and I.C.I., Ltd. E.P. 342,842. 12.2.31. Appl., 18.12.29.

Residues of solid matter consisting of carbon, ash, catalyst, etc., are obtained in the destructive hydrogenation of coal and the like.

A complete separation of the oils from these residues cannot be obtained by centrifuging or filtration and it is important to recover these oils. According to the present invention the oily residues are subjected to a counter current of heated inert gases while being mechanically conveyed in the form of a loose mass through a stationary retort, which is jacketed by hot gases.

The inert gas may consist of steam or flue gases or a mixture which can be heated to 500°—600° C. and which does not destroy the oils contained in the residue.

Oxygen or sulphuretted hydrogen should not be present in the gas used.

In one form of the invention the oily sludge is fed continuously from a hopper into a horizontal jacketed cylinder provided with an axial shaft carrying arms which stir the sludge and at the same time carry it through the apparatus. A counter current of steam and flue gas is passed through the cylinder and a bypass from the main gas supply connects to the jacket surrounding the cylinder in order to prevent loss of heat. The heating gas consists of superheated steam and flue gas in about equal proportions, and its temperature is controlled by addition of cool gas which has left the retort and from which the oils have been condensed. The gases in their passage through the retort carry off the oil in the vapour state from which the oils condense and the residue gases are turned to waste, excepting the portion which is recycled as mentioned. The material in the retort which has been deprived of its oil is withdrawn through a rotary valve or other sealed discharge and may be cooled in a drum from which it emerges in a dry granular form. As an example, 100 kg. of oily sludge was passed through the process and 28·5 kg. oil was recovered. Solvent extraction of the raw material showed 35·6 kg. of oil, so that the efficiency of the process is about 80%. Although the yield of the oil was not theoretical, the simplicity of the process rendered the cost per ton of oil recovered lower than by more elaborate methods.

The remaining residue can be used as a fuel or for making producer gas. Two modifications of an apparatus for carrying out the operation are shown.

R. P.

726. Low Temperature Distillation of Carbonaceous Material. A. Kath. E.P. 342,983. 12.2.31. Appl., 26.5.30.

A rotary kiln is described wherein a series of retort tubes are enclosed in a rotating drum so that the tubes are heated externally and internally by gases fed in at a rate automatically controlled by temperature measuring instruments. In a similar way the feed of carbonaceous material is controlled and a proper temperature gradient established in the kiln. H. C. R.

727. Distillation of Solid Carbonaceous Materials and the Manufacture of Gas. H. Nielsen and B. Laing. E.P. 342,817. 12.2.31. Appl., 22.11.29. Add. E.P. 292,060.

Lump coke is produced by a preliminary treatment at a temperature of 600° C. in an oxidising atmosphere. This product is then treated in a water gas generator. The gas obtained is treated in presence of a catalyst to form carbon dioxide and hydrogen, and after removal of the carbon dioxide the residual gas is utilised in methanol synthesis. H. C. R.

728. Low Temperature Carbonisation. J. Y. Johnson, Assr. to I.G. Farbenind. A.-G. E.P. 343,435. 11.2.31. Appl., 11.8.30. Add. E.P. 301,975.

The retort used is fitted with grates, which are permeable to gases, but not to the raw material, and are arranged in series one above the other. The hot gases are passed upwards and the charge is conveyed from one shelf to the next lowest by bucket wheels or similar contrivances. It is claimed that the separate processes of drying, gasification and degasification may be carried out independently on different grates. H. C. R.

See also Abstracts Nos. 640, 669.

ABSTRACTS.

Geology.

729. Individualism of Orogenies Suggested by Experimental Data. T. A. Link. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 385–403.

This paper constitutes a general discussion of the individualism of orogenic units as emphasised by experiments carried out by the author. The apparatus consisted of a pressure box fitted with jack screws which forced a concrete push-block against artificial strata. The face of the block had bulges and depressions for producing varying amounts of compression in the horizontal plane. A flexible rubber sheet was stretched across the side of the box against which the face of the block was to be forced, this side being supported by a removable board during deposition of the sediments. The artificial strata having hardened sufficiently, the board was removed and the push-block forced against the side by the rubber sheet.

Two experiments are described, and the results show that two quite different uplifts occurred by making a very slight change in the artificial sediments employed; one of these uplifts had features comparable with typical Foothills structure in Alberta.

Among the conclusions reached by this experimental work are: (1) Slight variations of factors or conditions which influence growth of structural units may cause extremely diverse results; (2) overthrust faults invariably develop at depth near the push-block (the source of the stress) and exhibit less and less displacement away from the block towards the surface; (3) underthrust faults develop at the surface near the block and show less and less displacement away from the source of stress.

The paper is very fully illustrated with tables and by photographs of the actual experiments described. G. S. S.

730. Stratigraphy of Permian Beds of North-western Oklahoma. N. Evans. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 405–439.

Much confusion appears to exist regarding the stratigraphical position of the Blaine and younger Permian beds, and discussion is here given on these formations of North-western Oklahoma with reference to adjoining areas.

A new classification is presented by the author, who includes all beds from the Blaine upwards under five formation names: Blaine, Dog Creek, Whitehouse, Day Creek and Quartermaster; the names Hackberry and Big Basin being dropped.

The Blaine formation is closely allied to the Dog Creek, both being made up largely of beds of gypsum and gypsiferous shale. The Whitehouse consists of three members, in ascending order: Marlow, Rush Springs and Cloud Chief. The lower two are characterised by limestones, dolomites and sandstones, and the upper (Cloud Chief) by massive gypsum embedded in red shales and sandstones. The Day Creek formation consists of dolomites which are somewhat lenticular, in places becoming sandy and grading into sandstones; these dolomites can be definitely placed by the sequence of beds and the change from the Whitehouse formation to the Quartermaster.

Above the Day Creek all the Permian beds of Oklahoma and Kansas are classified as belonging to the Quartermaster formation. The topmost beds of the latter are conformable with the Day Creek, and at no place in the Kansas and North-western Oklahoma area has any evidence of an unconformity been noticed, either at the base of, or within, the Quartermaster. Moreover, no deposits of gypsum have been observed anywhere in this formation.

Vertical and cross sections of these beds, together with a full bibliography, accompany the paper. G. S. S.

731. Oil Field Waters of Pennsylvania. C. F. Barb. *Penns. State Coll. Bull.* (*Min. Ind. Exper. Stn. Bull.* 8), 1931, 1–34.

In this report the author tabulates the results of many waters used for flooding in the Bradford and Alleghany districts, together with analyses of samples of salt water produced with the oil from wells occurring throughout the State. An attempt is made to show how these analyses may be simplified in many cases and the desired information still be obtained.

Discussion is also given to the possible mechanical relations of solutions, of oil-field waters and brines, and of oil-sands. In the plugging and cementing of oil-sands it is shown that the effect of iron hydroxide, silica and other sediments is much greater on coarse permeable sands of the Kane and Oil City regions than on those of the average Bradford type.

The investigation also proved that the analyses of flood waters do not show any average composition that could be selected as representative.

G. S. S.

732. Oil and Gas Prospects in Central Saskatchewan. P. S. Warren. Geol. Surv. Canada. Sum. Rept., Pt. B., 1929, 40–47.

The area is underlain by a horizontal series of Upper Cretaceous sediments, chiefly shales and sandstones. Owing to a heavy covering of glacial drift (20-200 ft.), exposures are rare over most of the district. The formations are, in ascending order: Belly River and Bearpaw (each estimated at about 400 ft.), and various fresh-water beds composed of sands, shales and clays (50 ft.) which have been grouped together.

The development of the oil and gas fields in Turner Valley and at Wainwright (Alberta) has created great interest in prospecting, though so far little success has resulted by drilling. Some of the more important of these prospects are described in detail. G. S. S.

733. Bituminous Materials in France (Occurrence of). V. Charrin. *Rev. Petrolifère*, 1931, **422**, 557–559.

In France there are many indications of the presence of bituminous material, but it would seem that only in very few cases can the deposits be economically used at present. The Soult-sous-Foret district has been exploited for many years, and by using deep borings and pumps a light oil rich in gas was obtained. By reverting to underground exploitation, the yield has been much increased. The oil which oozes from the sands flows in special channels to the wells from which it is pumped as in borings. The crude has the following properties: S.G. 0.870—0.960, up to 150° –10%, to 300° –25%, and paraffinous residue 65%. About 71,000 tons of crude oil were produced in 1929.

Other districts not at present worked are mentioned with some details of the geological nature of the oil-bearing strata. Shale deposits occur in many places, but while a small output was obtained in 1929 from the Autun district, shale-oil cannot compete with imported petroleum. C. C.

Field Technology.

734. Proration Develops Combination Head for Flowing Wells. F. Swindell. *Oil Weekly*, 1931, **61** (4), 47.

In the Oklahoma City Pool proration has been the means of introducing novel flow connections. The general practice has been to open wells to capacity and secure allowable production in a relatively small part of the month, even though this method is not as efficient as continuous flow. Wells have been allowed to flow through large strings of casing, resulting in high gas-oil ratios and correspondingly low lifting efficiencies. As a consequence production has declined in some cases at a rate of over 10% per month, and pressures have dropped in a like manner, and it has been evident that something must be done to prevent the necessity of placing all wells on the pump or installing gas lift.

Several operators have resorted to flowing wells through smaller strings of casing to reduce the gas produced per barrel of oil, and to maintain a slight back pressure on the sand.

Two principal objectives must be aimed at in designing an efficient flow connection if wells are to be so equipped that maximum production can be obtained in the short time allowed for taking potential tests. An arrangement must be made whereby the well can be produced at maximum rate for a limited time and additional connections must be provided for continuous flow, while securing production allowable under the specified percentage of potential.

With the hook-up used by the Deep Rock Oil Corporation the well may be produced wide open or it may be pinched to a few barrels per hour or a few drops. A 6 in. flow line has been welded into the 8 in. casing below the derrick floor and tubing run into the well under pressure. The 6 in. runs directly to the separator, and is so constructed that all right angle turns have been eliminated by the use of welds at obtuse angles. This promotes piston type flow by decreasing friction and tends to reduce back pressure, which results through decreasing the flow from 8 in. in the well to 6 in. above the derrick floor.

The hook-up consists of the 8 in. flow string with a tubing head fixed just above the derrick floor. Immediately above the tubing head is a 6 in. gate valve. In the 6 in. flow line is a 6 in. gate and check valve. Tubing is run into the well through the 8 in. flow string, and is provided with two 2 in. valves "A" and "B" above the 6 in. gate. A tee connection is made below the upper of the two 2 in. valves. One side of the tee passes through a valve "C" to a flow bean and thence to the separator line, whilst the other side joins a 2 in. flow line, which passes from the main tubing to the separator and is equipped with a valve "D."

When potential tests are to be taken the tubing is shut in by closing valve "A." After maximum flow has been obtained and the well potential established, valve "A" is opened and the 6 in. gate in the flow line is closed. The well is then in condition to be produced at any desired rate. With valves "B" and "C" open and "D" closed the flow is controlled by the flow bean. If it is necessary to remove the flow bean, valve "D" can be opened and "C" closed.

A bull plug is fitted to the top of the column of 2 in. tubing, and in the event of the well ceasing to flow this plug can be removed so that a swab may be introduced. L. V. W. C.

735. Gas Influence on Deep Well Pumps. A Fedoseev. *World Petr.*, 1931, **2**, 292.

Among all the factors which influence the working efficiency of the deep well pump, the influence of gases present in the well are the most important and have been the least studied.

Tests were made by the Azneft Oil Trust in Russia, and the results have shown that the working efficiency of the pumps is mainly influenced by the free gases, the appearance of which at the wells is caused by lowering the liquid level in the well or through the presence of a gas dome.

A study was made of both forms of gas influence and the results are given separately. From experiments made with various grades of oil it has been shown that the tendency is for oils to absorb all kinds of gases including hydrocarbon gases. It has been established that 0·001 m^3 of oil of 0·88 sp. gr. at 43° C. and at a pressure of 9·6 atm. will absorb 0·004 m^3 of gas. Assuming that at a pumping depth of 100 metres there are no free gases, as they have been completely dissolved in the oil, the pressure of the well at this depth will be :—

$$P_{abs.} = \frac{ha}{10} + 1 = \frac{100 \times \cdot 86}{10} + 1 = 9{\cdot}6 \text{ kg/cm}^2.$$

where a = sp. gr. of the oil
h = pumping depth.

When the pump is lowered into the well to its extreme position the volume of dead space is shown to be 0·005 m^3. This space will be filled with oil in the pipe at the same height, 100 metres. During the upward movement of the plunger the liquid enters the pump, and when the plunger stroke equals 0·457 metres the quantity of oil will have a volume of 0·0011m^3. On the downward stroke of the plunger the receiving valve closes, and the discharge opens and the plunger passes the liquid which is immovable.

If the hydraulic resistance caused by the oil under the plunger at its upward stroke, and the loss of pressure due to imparting of velocity to the liquid are disregarded, no gas separation would occur at the upward stroke of the plunger. The upward stroke of the plunger will, however, cause a decline in the pressure underneath by an amount which can be assumed to be 0·5 kg/cm^2. Thus the oil present in the dead space, and the oil coming from the well level, will separate gases due to loss of pressure. The volume will be determined by the difference between the volume of gas dissolved in the oil at 9·6 and 9·1 kg/cm^2. This volume will be lowest when the depth of the pump is greatest and when the dead space is smallest.

At the downward stroke of the plunger the pressure will increase, and when this pressure reaches 9·6 kg/cm^2 the separated gases will again dissolve in the oil.

The influence of free gases may have the effect of reducing the co-efficient of the filling of the pump to practically zero. When the plunger begins its upward movement, the suction valve will open and allow liquid and gases to pass into the pump cylinder. These gases are mixed with the liquid and flow in the form of bubbles, and on passing the pump filter they are dragged along into the pump cylinder. This occurs when the velocity of the gases is lower than that of the liquid.

Tests were carried out with pumps of various types and sizes, and results have shown that with a gas anchor attached to the pump, a co-efficient of filling can be obtained up to 0·74, whereas with no gas anchor the co-efficient may be nil.

From these tests it has been concluded that the presence of dissolved gases in the liquid has little influence on the working efficiency of the pump, but that free gases are highly detrimental. L. V. W. C.

736. Method Used for Setting Longest String of 13¾ in. Casing. R. E. Leigh. *Oil Weekly*, 1931, **61** (4), 29.

A string of 13¾ in. casing over 1¼ miles long has been successfully landed and cemented in a well in the Kettleman Hills district. This unusually long string was necessary, as the well is located well down the flank and production is expected at around 9000 ft. The key beds have not yet been found, and it is likely that the ultimate depth will reach 10,000 ft.

The calculated weight of this string is about 459,884 lb. The weight was suspended by a 300 ton hook, a five sheave travelling block, and a 1 in. by 2500 ft. superstool casing line under a 136 ft. by 26 ft. derrick made of special high tensile steel and reinforced with 5 in. reinforcing legs, and an 8-sheave double deck crown block. The manufacturers estimated that the capacity of the derrick exceeds the weight of this string by more than 50%. In this case the weight of the string was relieved by about 25% by the use of two cement float collars which allowed the pipe to be floated into the hole. One float valve was placed on the bottom of the string, and the second was placed in the first collar above the shoe as a precautionary measure. A cement whirler shoe, with side ports for the establishment of circulation when on bottom, was also used.

The casing was landed on a spool type casing head, and after it was set and cemented a stretch was taken of practically the full weight of the string. The casing was then packed and cut off flush with the head.

The string was run in to a depth of 6766 ft. in under 19 hours, and was lifted back to 6763 ft., where it was cemented by the Perkins method with 850 sacks of cement, half of which was special oil well cement and the other half ordinary construction cement. It required 1½ hours to pump the second plug down, and it took 775 lb. to land it. Circulation broke at 350 lb., and was maintained at 200 lb. When the plug was drilled only 38 ft. of cement was found inside the pipe.

Conductor pipe of 18⅞ in. diam., 84½ lb., was set in 27 in. hole at 1381 ft., and the hole was then reduced to 17½ in. until the depth of 6766 ft. was reached. The pipe was supplied in 42 ft. joints, thus materially reducing the number of joints to be made up and the time required for running.

The hole was surveyed repeatedly and the maximum deviation from perpendicular was found to be less than 2°, the average being something less than 1°.

The well was drilled with a hydraulic rotary outfit, and the weight on the bit was constantly maintained and recorded. The equipment included a string of 6 in., 25 lb. special alloy high tensile drill-pipe, high pressure pumps and a variety of patent bits. L. V. W. C.

737. Oil Well Service Steel Requirements. H. T. Morton and I. A. Rummler. *Oil and Gas J.*, 1931, **29** (44), 54.

There is a big demand in the oil industry for a stainless steel of maximum hardness and corrosion resistance consistent with high impact value, particularly for the balls and seats of pump valves, needle valves, etc. Such a steel is that covered by Armstrong's Patent, No. 1,322,511, of 1919, concerning which little information is published. Information concerning the corrosion characteristics and the proper heat treatment during the preparation of the alloy to avoid internal defects has been collected.

Metals of this nature fail either by erosion, due to foreign matter in the oil, corrosion due to acids or chemicals in the oil, or by flaking and cracking due to low impact resistance. Erosion is usually manifested by the appearance of worn spots, whereas corrosion usually produces circular pits and

long hairlines on the surface. These hairlines usually extend between the "poles" of the ball, while the pits are situated near the poles (the poles are the areas of the balls corresponding to the ends of the original round bars from which the balls are made), indicating that the cause of the pitting was an internal defect in the original bar and not in the surface of the steel. Flaking and cracking is also caused by poor heat treatment and forging, internal defects weakening the resistance to impact.

The composition of the alloy under consideration is as follows: Carbon, 1·10%; chromium, 17·24%; Manganese, 0·36%; Silicon, 0·52%.

Great care must be taken during forging and cooling operations to prevent the formation of internal defects.

The optimum temperatures for pre-heating, forging normalising, quenching and drawing are given with details of the precautions that must be taken in cooling.

The effect of the drawing and quenching temperature on the hardness of the final alloy on Rockwell C. scale units is also described. C. L. G.

Crude Oil.

738. Distillation of Hydrocarbons. I. C. Carpenter and A. R. Moorman. Assrs. to Contact Filtration Co. U.S.P. 1,797,715, 24.3.31. Appl., 17.9.26.

The apparatus described consists of a tank, a pipe still, means of supplying liquid from the former to the latter, and a separating chamber connected to the still. Connexion is made between the chamber and the tank, and a vent is provided for allowing the escape of vapours from the chamber. Means, controlled by the level of the liquid in the chamber, are provided for maintaining a constant volume of liquid in the tank. The body of oil is maintained at a temperature above the vaporisation point of water but not above that of a desired oil fraction. Vapours are separated from the heated liquid, the unvapourised portion being returned to the main body of oil, while a stream of oil to be treated is fed on to the surface of the heated body of liquid.

W. H. T.

739. Breaking Petroleum Emulsions. C. H. M. Roberts, assr. Tretolite Co. U.S.P. 1,802,090, 21.4.31. Appl., 24.1.27.

An inert gas is incorporated in the emulsion prior to subjecting the emulsion to homogenisation. Any suitable gas may be used, such as compressed air, at temperatures low enough to prevent oxidising reactions or carbon dioxide or natural gas. W. L.

740. Distillation Apparatus. F. G. Ring, assr. Petroleum Derivatives, Inc. U.S.P. 1,802,274, 21.4.3. Appl., 19.9.25.

In this still the oil flows continuously from the supply tanks through a preheater, a separator chamber, the main still, and so to the receiver tanks.

Heat is supplied to the oil in transit in sufficient quantities to cause vaporisation as desired. The conduits through which the oil travels are exposed to a current of combustion gases flowing in a counter-current direction to the oil. The vapours evolved are removed from the still at various points along the path of the oil stream and pass to condensing units. Vapours from the separator are removed by a vacuum pump and the liquid is withdrawn as rapidly as it collects and pumped through the main still. W. L.

See also Abstract No. 748.

Gas.

741. Chemical Detection of Leaks in Gas Distribution Systems. C. F. Turner, *Oil & Gas J.*, 1931, **29** (46), 141.

The detection of leakage in gas lines is facilitated and cheapened by employing men equipped with gas detectors and the necessary tools, &c. A systematic inspection of underground vaults, cable ducts, water vaults, sewers, curb boxes, etc., is carried out with the aid of the gas detectors, which record up to 3·5% of combustible gas, the lower explosive limit of a gas/air mixture. The men are also provided with a CO detector in order to ascertain whether natural or manufactured gas is leaking. Another crew obtains samples from the manholes, etc., reported by the street crews to contain an explosive mixture, and the exact percentage of combustible gas is determined. It is claimed that this system enables the exact location of the leak to be more readily ascertained. After repairs have been effected the manhole is again sampled. The routine employed is fully described. The introduction of odorants into natural gas has not proved eminently successful in detecting distribution main leaks, since all complaints received emanated from leaks beyond the meter and in the house lines. Odorants are, however, of value in detecting leakages in factories, where by injection of the odorant into the house line following the meter, leakages are readily detected and marked for repair.

R. A. E.

742. Butane-Air Gas Business. E. Kaye, *Petr. Eng.*, 1931, **2** (6), 68.

The development of casinghead gasoline plants and of refinery vapour recovery systems has led to the production of increasing quantities of butane, and attempts to apply this product in place of ordinary gas, both for industrial and domestic purposes. The largest offtake is provided by town air-gas systems, scores of which are now in operation.

Descriptions and flow sheets of the two most important types of butane-air gas plants are given. In one type the butane is vaporised, reduced to air temperature, mixed with air, and then compressed to the necessary delivery pressure. In this type the composition of the "butane" must be known to insure that the final product has the necessary calorific value. In the other type (the liquid carburation process) the air only is compressed and the liquid butane passed through, it being vaporised by the heat of compression of the air. Control is effected directly by determination of the calorific value of the final product. The advantage of this process is that any type of liquefied gas can be used, while economies are effected by the omission of the compression of the butane and by the utilisation of the heat of compression of the air to vaporise the butane.

A chart is given showing the heat of compression available to provide heat of vaporisation of the butane, and the discharge temperatures of the compressed air.

C. L. G.

743. Present Positions of the Thermal Decomposition of the Lower Hydrocarbons. A. E. Dunstan, *J.S.C.I.*, 1931, 50, 325–326.

The production of high anti-knock motor spirits from natural gas or "cracker" gases by heat treatment has received attention; use could also be made, with cracker gases, of the tendency of the olefin hydrocarbons to polymerise, producing low-boiling liquid hydrocarbons.

Paraffin hydrocarbons under conditions of pyrolysis form aromatic hydrocarbons, but several factors render large-scale work difficult. Paraffins require temperatures of 800–1050° C., with conversion of 22—27% of the

paraffins passed; on the other had olefines require a lower temperature 700—800° C., the conversion of olefines being 36–40%, the liquid produced being similar to that obtained from paraffins.

For the formation of olefines from paraffins, the pressure must be atmospheric or even reduced, owing to the increase in volume, and it would appear that olefines may be formed from paraffins by mild pyrolysis.

Polymerisation as applied to olefines involves a reduction in volume, the temperature required is lower than that required for pyrolysis (up to 550° C.), while pressure was found to be an advantage. The liquids obtained were not aromatic and yields approaching theoretical were obtained. The recorded 92% conversion was not a maximum, and of the total liquid 86% boiled below 200° C., the spirit having an engine test five-sixths that of benzene.

W. A. W.

744 Natural-Gas-Conversion Process. M. P. Youker. U.S.P. 1,800,586. 14.4.31. Appl., 1.5.25.

Hydrocarbon gas is converted into comparatively non-volatile hydrocarbon liquid by submitting the gas to a pressure greater than 500 lbs./sq. in. and feeding the compressed gas continuously through a heating zone maintained at a temperature above 750° F. The gas is kept heated and compressed until a portion is converted into liquid hydrocarbons; the resulting mixture is then separated into fixed gas, which is constantly released from the apparatus, gasoline which is withdrawn from the process and hydrocarbon vapours which are heavier than the fixed gas and lighter than the gasoline. These hydrocarbon vapours are returned for admixture with fresh gas. W. S. E. C.

See also Abstracts Nos. 749, 803, 804.

Motor Fuels.

745. Seasonal Gasoline Changes Practical. R. C. Alder. *Oil and Gas J.*, 1931, **29** (42), 144.

Based on the conclusions of other investigators charts are plotted to show the relation between the A.S.T.M. distillation of a gasoline, the minimum temperature at which starting is possible with an air/fuel ratio of 1 : 1 and a resultant air/vapour ratio of 20 : 1, and the minimum temperature at which easy starting can be obtained with an air/fuel ratio of 1 : 1 and a resultant air/vapour ratio of 12 : 1. The minimum air/fuel ratio necessary to provide easy starting at any given atmospheric temperature when using a gasoline of known volatility can also be deduced by means of the chart. It is demonstrated that practically all present-day commercial gasolines will give easy starting except under most extreme conditions. The "warming-up" characteristics, or performance of the gasoline during the first few minutes of operation, the rapidity of acceleration without loss of power and rate of crankcase dilution, have therefore become subject to closer scrutiny. These characteristics are governed by the portion of the A.S.T.M. distillation curve at which 35, 65 and 90% are evaporated. Curves are given to show the effect of car conditions and atmospheric temperatures on the gasoline properties needed for good performance. The whole of these effects are then combined on one chart and related to the percentages evaporated by A.S.T.M. distillation and to the distillation temperature. A diagram is added to show the maximum allowable Reid vapour pressure at the prevailing atmospheric temperature in order to avoid vapour-locking. The

distillation curve of the composite winter gasoline as reported by the U.S. Bureau of Mines for January, 1930, is plotted on the chart. It is seen that such a gasoline will efficiently operate cars fitted with automatic radiator shutters, an efficiently heated manifold hot spot and a carburettor fitted with an efficient accelerating device. On the other hand, when such gasoline is used in cars not so equipped, more than 2 min. is required to heat the manifold to the point where the choke is no longer required at atmos. temperatures below 50° F., and good acceleration without loss of power is only possible at temperatures between 50° and 100° F. By reducing the A.S.T.M. 65% temperature without changing appreciably the 90% temperature, the performance in the second type of car would be greatly improved in both respects without causing depreciation in the performance of the gasoline in the first type of car, always provided the vapour pressure is correct.

A table is presented showing the properties of 22 extreme and typical fuels marketed and estimates are made of their performance in both types of car. The table demonstrates that winter gasolines are now being marketed with seasonal limiting vapour pressures, high 90% temperatures and low 35 and 65% temperatures, and the cold weather performance of these gasolines is admirably balanced from all the points of view considered above. One company has instituted a scheme of controlled seasonal gasolines. Five grades have been devised to provide a good all-round performance, judged by the preceding methods, at atmospheric temperatures of (1) 45° F. max., (2) 45/65° F., (3) 65/75° F., (4) 75/85° F., (5) over 85° F. The maximum vapour pressures (Reid) are 13, 11·75, 10·5, 9·25 and 8 lbs. respectively. From a study of weather reports the most suitable grade for use in each state for each month of the year has been estimated and a table is presented showing the monthly distribution plan which has been adopted. Each refinery of the company has its own natural marketing territory, and it has generally been found possible to operate the plan throughout the whole country without keeping more than two grades in stock at any refinery at any particular time. R. A. E.

746. Fuel Research at Bureau of Standards. O. C. Bridgeman. *Oil and Gas J.*, 1931, **29** (42), 143.

The Bureau of Standards has for a number of years been working in co-operation with the automotive and petroleum industries on the problem of the relation between fuel characteristics and engine performance. The general procedure has been (1) To ascertain fundamental variables involved, (2) The investigation of independent effect on each variable in laboratory apparatus, (3) The development of practical tests for fuel properties, (4) The establishment of significance of these tests by engine work, (5) Study of effect of engine design on the permissible values of the fuel property investigated. The more significant fuel properties are :—

(1) Volatility in respect to engine starting, acceleration, distribution and crankcase dilution. It has been found that the A.S.T.M. distillation curve of % evaporated (*i.e.* distillation loss added to recovery) is a practical measure of fuel volatility. Assuming a normal engine in good mechanical condition, starting should not be difficult until temperatures drop to 140° F. below that of the 10% A.S.T.M. evaporation temperature at a fuel/air ratio of 1 : 1. Ease of acceleration is related to that portion of the A.S.T.M. curve covering 30/70% evaporated, and distribution to the portion 10/90% evaporated, the lower the temperatures the better the acceleration and, up to a point, the distribution. Manifold design has a great effect on both properties but not on the relative value of various fuels tested. The 90%

evaporation temperature is a criterion of dilution, and also a measure of the maximum temperature to which the air/gasoline mixture should be heated in the manifold.

(2) Vapour pressure in respect of gas lock. For automobiles the main factor is the temperature at which the vapour pressure becomes equal to atmospheric pressure. It has been found that (*a*) Reid vapour pressures at 100° F. × 1·1 = gas-free vapour pressures at the same temperature; (*b*) the A.S.T.M. 10% evaporation temperature is within 2° F. of temperature at which the gas-free vapour pressure equals atmospheric for propane-free gasolines; (*c*) I.B. Pt. determined by thermometer in liquid is also approximately equal to this temperature but is difficult to determine. By experiment it was found that for both airplane and automobile fuel feed systems the vapour-locking temperature t in °F. is given by the formula

$$t = 560 \frac{(5{\cdot}167 - \log.\ 1{\cdot}1PR)}{(5{\cdot}167 - \log.p)} - 458,$$

where PR = Reid vapour pressure and p = atm. pressure at the given altitude, both in lb./sq. in. A discussion of the effect of various fuel systems on vapour locking follows, and maximum safe vapour pressure limits for various average driving conditions are given. Propane should be removed particularly from aviation fuels.

(3) Gum content. The work on this subject is incomplete, owing to present lack of an adequate method of determination.

(4) Sulphur content. Corrosion of the fuel feed system is caused by corrosive sulphur compounds. Total sulphur content appears to be of major interest for crankcase corrosion, but the generally accepted limit of 0·1% is based on limited information and is probably low especially for warm climates. An investigation is projected.

(5) Antiknock value. This problem is being investigated by the Bureau and by the C.F.R. Steering Committee. The adoption of a standard primary reference scale and of octane nos. has assisted in clearing up past discrepancies between laboratories. A tentative standard test engine has also been developed and engine variables are being studied. R. A. E.

747. Dyes to Reduce Loss in Acid Treatment. G. Egloff, J. C. Morrell and P. Truesdell. *Oil and Gas J.*, 1931, **29** (42), 133.

The colouring of gasolines is usually done with a view to their ready identification or for masking any discoloration present. Blue and violet dyes are also used in smaller proportions to neutralise any coloured compounds present, thus giving the gasoline a water white appearance. The necessity of acid refining a slightly yellow gasoline can be avoided by judicious dye treatment, thus avoiding the loss of anti-knock rating as well as the usual refining losses resulting from acid treatment.

It is calculated that 25% of the motor fuel sold in the U.S.A. is artificially coloured, the increased use being due to the increased amount of cracked gasoline sold, the refining of which to a water white colour is expensive.

In the dyeing of cracked gasoline care must be taken to select a suitable dye since many of them turn quite different colours when added to the gasoline. Financial savings can be effected by the use of a blue dye for giving an unrefined cracked gasoline a white colour, the cost of the dye being about 0·4 c./bl. as compared with 8 c. for acid refining to a water white colour. Dyeing is effected by adding sufficient of a solution of the dye in benzol to give the gasoline a slight bluish tinge when examined in a colorimeter, but a water white appearance to the naked eye in globes, etc.

A considerable amount of work has been carried out at the Riverside laboratories of the Universal Oil Products on the effectiveness of various dyes on Pennsylvanian cracked gasoline and Californian straight run gasoline. Tests were carried out by adding 2 mg. of dye to 100 ml. of gasoline, filtering off any excess and exposing the solution to sunlight for 50 to 60 hrs., any fading, precipitation, etc., being noted. A number of dyes were found to be insoluble in gasoline, though all were soluble in benzol, only those soluble in gasoline being tested further. The quantity of dye to be added was determined by matching a solution of the dye against an arbitrary colour standard colour.

It was found in general that yellow and blue dyes were stable in both types of gasoline, while solutions containing less than 2 mg. per 100 ml. gasoline were less stable than more concentrated solutions.

Full results of the tests on the various dyes are given. C. L. G.

748. Gasoline from Signal Hill Crudes. O. C. Smith, *Oil & Gas J.*, 1931, **29** (42), 136.

Signal Hill Crude contains approx. 1½% sulphur and yields 28/30% of motor gasoline. About 50% of this gasoline has a good colour, is fairly colour stable, and gives a negative copper strip test. The remainder is quite unstable, probably due to the presence of hydrogen sulphide and free sulphur. The first 50% is therefore obtained directly from the evaporator, the bottoms from which are pumped to three stills in parallel, where the remainder of the gasoline is removed and pumped into one tank. After three hours' storage in the dark the colour of gasoline from the stills drops from 27 to 22 Saybolt. It has been found that a caustic wash prior to storage considerably reduces the quantity of acid necessary to obtain a product having a good copper test and colour stability. When the caustic wash is omitted it is thought that the acid and H_2S react to form free sulphur, which it is difficult to remove by the use of acid. Data showing the beneficial effect of the caustic wash is presented. On the plant the acid treatment is carried out continuously, sludge acid from the first tower being introduced into the suction of the circulating pump. The acid sludge is used until the gravity drops to 43/45° Bé, when some fresh acid is added and some sludge drawn off to keep the acid level in the tower constant. The last traces of sludge are removed from the gasoline by settling and passing through towers packed with granite rock of varying sizes. Caustic and water washes are given before the gasoline passes finally to storage. A flow chart of the treating process is given. R. A. E.

749. Use of Refrigeration in Gasoline Plants. F. L. Kallam, *Oil & Gas. J.*, 1931, **29** (43), 103.

A history of the development of the various types of casinghead gasoline extraction plants is given, with particular reference to the use of refrigeration. The extraction of gasoline from gas may be said to date from the year 1903, when casinghead gasoline from Sistersville W. Va, was used for boiler fuel. In the winter gasoline condensed in low spots in the line and was bled off and sold; eventually coils refrigerated with cold running water were installed and a permanent supply of gasoline insured. Gas compressors working at 250 lbs. came into use in 1905, using many variations of water-cooled devices, followed by expansion into low-pressure systems. The Heinzerling patent of 1897 was not employed till 1906, condensation being effected by a compressor and an expansion cylinder coupled to the same flywheel, this system being developed until, in 1920, practically every compressor plant in California had its expander units, though they were not used to the same extent in other areas. Much trouble was experienced owing to the difficulty of lubrication

at low temperatures and to the freezing of moisture within the machines. This was got over by using a turbine instead of a reciprocating steam unit. The next stage in development was cooling by means of evaporation under a partial vacuum of gasoline produced at high pressure, working on the same principles as an ammonia compressor, the evaporation taking place in a heat interchange handling the wet incoming gas, the temperature of which is lowered by the evaporation of the lighter fractions. Direct refrigeration by means of ammonia, or calcium chloride and brine cooled by ammonia was also used.

In 1918 the first refrigeration-absorption plant was built, being originally designed for direct cooling, but owing to freezing and discoloration troubles the cooling medium was changed to naphtha. The gas was allowed to flow through towers over which was showered naphtha cooled to 35° F. by ammonia. The enriched gasoline was mixed with the absorbing naphtha and the excess, not required for recirculating run to storage. In the plant the extraction was said to have been 0·26 gal. per 1000 cu. ft. gas. In 1919 similar plants were constructed, mineral seal oil being used and the absorbed gasoline removed by steam distillation. In later years the use of refrigerants declined, chiefly owing to the cost of their production.

In 1921 the first commercial gasoline rectifier unit was built, operating at low pressure, and accordingly requiring a low temperature at the top of the column. The vapours leaving the column were recompressed, cooled and condensed, and a proportion introduced into the column and expanded from the condensing pressure to that of the column. This expansion, together with the heat provided by the kettle at the bottom, provided the necessary temperature gradient for rectification. The temperature of the vapours at the top was of the order of 10° F., probably corresponding to a column pressure of 8 lbs. These low temperatures caused such difficulties in operation that the low-pressure system of rectification was abandoned.

In 1926, S. C. Carney designed a stabilising column, using a narrow cut product from natural gas (boiling between −50° F. and 40° F.) as refrigerant. The vapours from the stabiliser were compressed to 175 lbs., condensed, and a proportion expanded to 20 lbs. through the shell of an oil cooler, the resulting vapours being recollected and recompressed. When sufficient refrigerant is obtained the system becomes a closed cycle, except for the make up for losses. The absorption oil was finally cooled to a temperature of 50° F., close control being effected by manipulating the pressure at which the refrigerant was allowed to evaporate in the cooler, and by regulating the amount of tube surface bathed in the evaporating liquid. This plant was entirely satisfactory from the commercial standpoint.

In recent years further attempts have been made to use refrigerated brine to cool the gas by direct contact, but such plants were only economical when used with rich gases. During the past year a modification of the Carney method has been used in cooling Kettleman Hills gas, using the overhead product from the stabiliser (principally propane) and evaporating in a gas cooler at a pressure of 100 lbs. and a temperature of 58° F. The vaporised refrigerant is compressed to 165 lbs., stored in an accumulator, and evaporated at 160 lbs. at 84° F. Five million cubic feet of propane per day are used to cool 60 million cu. ft. of wet gas through a temperature range of 35° F.

The use of a propane as refrigerant allows the use of very low oil to gas ratio in the absorber intercooler, a ratio of 1 : 5 giving a finished product equal to that obtained formerly with a 1 : 8 ratio. Considerable economies are thus being obtained by the use of propane for cooling absorption oil, particularly in the tropics, where the temperature of cooling water is high. Further development in the use of refrigeration is expected. C. L. G.

750. Resolution of Hydrocarbon Vapours. F. A. Howard and N. E. Loomis, Assrs. to Standard Oil Dev. Co. U.S.P. 1,797,145, 17.3.31. Appl., 25.10.27.

Gasoline vapours are subjected to partial condensation in order to produce a first condensate requiring chemical treatment, and a second vapour which requires no chemical treatment and has an e.p. of approximately 300°–350° F. The second vapour is separated into a liquid product free from gaseous hydrocarbons and into a gas. The first condensate is treated and blended with the liquid products from the second vapour. W. H. T.

See also Abstracts Nos. 743, 744.

Lubricants and Wax.

751. Alignment Chart for Estimating Viscosity-Gravity Constant of Petroleum Lubricating Oils. W. F. Houghton and J. A. Robb. *Ind. Eng. Chem., Anal. Ed.*, 1931, **3** (2), 144.

The viscosity-gravity constant as developed by Hill and Coats has been found of use in classifying lubricants. It affords a convenient means for following the progress of various refining processes such as acid treatment and solvent extraction.

The viscosity-gravity constant is based on a relation between Saybolt viscosity and specific gravity thus :—

$$G = A + \frac{1{\cdot}0752 - A}{10} \log (V - 38)$$

where A = viscosity-gravity constant, G = sp. gr. at 60° F., V = Saybolt viscosity at 100° F.

When the viscosity is measured at 210° F. the equation is $9 = 0{\cdot}24 + 0{\cdot}755A + 0{\cdot}022 \log (V^1 - 35{\cdot}5)$. These equations are cumbersome in use, and so an alignment chart is given which permits the rapid estimation of the constant from the viscosity and gravity data. W. L.

752. Lubrication from Engineering View. A. C. Mougey. *Oil and Gas J.*, 1931, **29** (44), 30.

Tests have been made in an engine operated at 45 m.p.h. to determine the effect of viscosity of the lubricating oil at 210° F. on its consumption, which generally determines the minimum viscosity to be used. The results are presented graphically and show that there is an optimum viscosity for minimum consumption, which will naturally vary according to the engine and operating conditions. Increased consumption with oils of lower viscosity is due to increased leakage past the bearings to the cylinder walls and with oils of higher viscosity is due to decreased ability to drain from cylinder walls or to escape through oil holes. A difference in consumption noted in certain oils having similar viscosities is due to variations in the volatility of the oils. Consumption of oil is greatly influenced by the speed, the use of oil control rings, and of oil coolers, and all these factors must be taken into account in selecting the most suitable viscosity for the lubricant to be employed. Maximum viscosity is even more important on account of the starting difficulties attendant upon the use of an oil too high in viscosity. The viscosity of an oil at low temperatures is as important as the pour test in determining the flow of oil to the pump inlet under winter conditions. Tests have shown that unless the starter can crank the engine at 25/50 revs. per min. starting

is difficult. Three oils, S.A.E. 30, S.A.E. 10 and a special light oil, were examined in a 6-cyl. engine, using a fully charged cold battery, and the minimum temperature at which the starter gave 25 and 50 r.p.m. of the crankshaft determined. These results, together with the oil viscosities at temperatures ranging from −20° to 210° F. are given, and show that the lower the oil viscosity the lower the minimum starting temperature.

In actual practice, starting at temperatures lower than those determined for each oil are obtained on account of the dilution of oil with gasoline. For example, a 10% dilution will practically halve the viscosity of an oil originally 300 sec. at 100° F., so that the use of prediluted oils is a possible solution for starting difficulties encountered in winter after changing oil. Another solution is the use of oil temperature regulators, which assist the rate at which the oil is heated initially, and also, by reducing the temperature of the oil under high speed driving conditions, allow the use of a thinner oil which in its turn facilitates starting.

The property of "oiliness" is discussed. Generally speaking, it is difficult to show any advantage due to this property, since automobile engines operate so well under normal conditions on oils which do not possess more "oiliness" than regular commercial motor oils. There are, however, a few exceptional instances where oils possessing superior "oiliness" are necessary. The method employed for determining "oiliness" in the laboratory of the General Motors Corporation is described and results obtained on certain oils and greases shown.

The S.A.E. system of classifying engine transmission and rear axle lubricants is described and commended for general application on account of the assistance which it renders to the purchaser in selecting the oil possessing the most suitable viscosity for his purpose. The stability of oils during use is important, but tests to measure stability should be governed by a consideration as to whether the changes indicated interfere with the suitability of the lubricant for the purpose for which it is intended. Otherwise application of such tests may restrict the supply and increase the cost of lubricants. R. A. E.

753. Some Problems of Diesel Cylinder Lubrication. E. H. Hillman *Petr. Eng.*, 1931, 2 (6), 186.

Diesel cylinder lubrication should be considered from 3 angles: (1) Cleanliness of the oil—*i.e.*, its resistance to carbonisation around the pistons, etc., and to sludging if the same oil is used in the crankcase. At present service tests are necessary to evaluate oils in respect of cleanliness but uniformity of operating conditions is essential. (2) Rate of cylinder and ring wear, which is important on account of replacement costs and the loss of power and the deterioration of oil. Tests of about six months duration are necessary to obtain reliable information on cylinder wear owing to the difficulty of making accurate measurements. It appears that in some instances a moderate increase in viscosity over typical recommendations would be beneficial, especially now that improved methods of manufacture make it possible to obtain lubricants of higher viscosity without increased carbon forming characteristics. The belief that a moderate increase in oil viscosity decreases wear has not been definitely proved by tests, and the subject requires further investigation. An increase in viscosity will also tend to decrease consumption. (3) Power loss from friction, which amounts to 15/25% of the i.h.p. Fluid friction is proportional to the viscosity of the oil, hence an oil of the lowest viscosity for satisfactory operation and rate of wear should be used. In one test a reduction in viscosity at 100° F. of 500 sec. caused a decrease in fuel consumption of 3·7%, other conditions being similar.

The carbon residue test has been proved to be of no value in evaluating carbon and gum formation tendencies in the engine, except possibly when comparing oils from the same crude, and no other laboratory test has yet been proved to give satisfactory results. Pour point is of no significance in the majority of Diesel installations, and flashpoint is not believed to be a useful guide to volatility and hence to oil consumption. The test of prime significance is viscosity at 210° F., the highest commercial temperature employed and most nearly approaching temperatures reached in the upper cylinder, which is the significant point from the lubricating standpoint.

It is suggested that engines should be divided into 4 classes: (1) 2 cycle, no piston cooling; (2) 2 cycle, with piston cooling; (3) 4 cycle, no piston cooling; (4) 4 cycle, with piston cooling. Since the higher the M.E.P., the higher the average temperature to which oil is subjected on the cylinder wall, and since for the same type of piston the larger the diameter the higher the temperatures experienced on piston walls and rings, the strain on the lubricating oil should be proportional to M.E.P. × D, where D = diam. of piston. Assuming frictional losses proportional to B.M.E.P. for convenience of calculation the factor B.M.E.P. × D may be used. It is therefore suggested that carbon formation, friction loss, wear and consumption should be accurately measured in a variety of engines, running under full load conditions using oils of varying viscosities and the results compared with the factor B.M.E.P. × D. Pre-supposing the establishment of a relationship for each class of engine, the selection of the most suitable viscosity required to meet any given conditions would be facilitated. R. A. E.

754. Production of Hydrocarbons of High Boiling Point. J. Y. Johnson and I. G. Farbenind A.-G. E.P. 345,334, 18.3.31. Appl., 18.12.29.

When unsaturated hydrocarbons are, by heating with aluminium chloride, converted into hydrocarbons of higher b.p. which are capable of being worked up into lubricating oils, there is a tendency for cracking and for the formation of asphaltic products. This objection is obviated by effecting the condensation of the hydrocarbons of low b.p. in the presence of the double compound of ethylene with aluminium chloride.

The reaction temperature and the duration of the condensation depend on the nature of the initial material. Usually good results are obtained at temperatures varying between 100° and 180° C. The higher the iodine value or the lower the molecular weight of the initial material, the greater must be the duration of the treatment and the amount of condensation agent employed. Examples are given of the polymerisation of isobutylene, "cracked" paraffin oil (iodine value 140), brown coal tar oil, etc. The reaction products are completely distillable, and the boiling range obtained is given in each instance. W. D. S.

755. Recovery and Purification of Lubricating Oils. Indian Refining Co. E.P. 345,632, 16.3.31. Appl., 18.10.31.

A process for manufacturing lubricating oils from a wax-bearing fraction obtained by distillation of a petroleum oil at low pressure is described. A selective solvent such as a mixture of benzol and acetone in equal proportions is added to the said fraction. The resulting solution is cooled to a temperature below 0° F., and the wax removed. Certain conditions govern the selection of the solvent. It must consist of a liquid having complete solvent action on the fraction employed, at 90° F., no solvent action on the solid components at temperatures below −5° F., and of such a nature that on removal of the solid hydrocarbons and solvent, the resulting oil has a cold test substantially

the same as the temperature at which the solid hydrocarbons are removed. Treatment of the wax-bearing fraction with acetone prior to the treatment with the selective solvent is suggested as a modification of the process. After the dewaxing, the fraction is distilled at reduced pressure. An apparatus is described. W. D. S.

756. Methods of Purifying Oils. British Thomson-Houston Co. E.P. 345,708, 2.4.31. Appl., 9.1.30.

When insulating oils are subjected to high temperatures, chemical influences and dielectric stresses, chemical and physical changes occur in the oil which depreciate its insulating properties. Some of these changes are caused and promoted by the catalytic effect of metals, particularly copper. A method for retarding these changes is described. A charge of glycerine or other equivalent polyhydric alcohol is maintained in contact with the oil, but out of contact with the insulation and electrical members of the particular device employed. Thus in a transformer comprising a core and windings immersed in a charge of insulating oil and contained in a tank but supported clear of the bottom of the tank, glycerine (an equivalent polyhydric alcohol) is placed at the bottom of the tank out of contact with the insulation and electrical members of the transformer. An absorbent material may be used with the glycerine.

The polyhydric alcohol prolongs the useful life of the oil. It appears to function as a neutral solvent extracting impurities formed in the oil during use, continuously. It also acts as a retarder of chemical changes, colour, sludge and acid formation of oil heated to a high temperature in the presence of copper. W. D. S.

757. Purification of Lubricating Oils. R. A. Wischin. U.S.P. 1,792,882, 17.2.31. Appl., 10.10.28.

The oil to be purified is intimately mixed when cold with concentrated sulphuric acid, and the mixture is gradually heated during the course of several hours to a temperature of about 50°–60° C., whereupon it is filtered after flowing through a sludge separator of known type, and then flows through an absorbing agent, such as fuller's earth, and finally has steam injected into it. By mixing the oil and acid in the cold and gradually warming polymerisation is avoided. The steam removes the fuel diluent.

H. E. T.

Fuel Oil.

758. Combustion of Fuels. R. Zaniroli. E.P. 341,052, 6.1.31. Appl., 6.8.29.

The present invention provides improvements to a method of combustion detailed in E.P. 278,374 of 1926. Fuel is admitted to a trough or series of interconnected troughs, and on burning heats water in a boiler situated above it. The steam so produced is projected horizontally through holes in the boiler walls at such a height as to impinge normally to the flame at its hottest portion. The steam dissociates into hydrogen and oxygen, the former burning to produce heat and the latter increasing the combustible capacity of the air. The fuel and water supplies are regulated by cocks in the supply lines, and admission of air is controlled by two dampers. Supplementary air is admitted by another damper, and issues through a series of holes above the boiler to complete the process of combustion.

J. M. F.

759. Oil Fuel Burners. Shell-Mex, Ltd., I. Lubbock and J. R. Joyce. E.P. 341,486, 16.1.31. Appl., 16.10.29.

This relates to automatic burners in which ignition is intermittent and provides means for cleaning the electrodes of an electric spark igniter. The electrode wiper is moved by a member subjected to the pressure of the oil fed to the burner. The same member is also utilised to operate a vane or damper for controlling the air supply to the burner, and if desired can be made to control the valve supplying oil to the burner nozzle.

W. L.

760. Oil Fuel Burners. J. W. Jones. E.P. 342,363, 5.2.31. Appl., 5.10.29.

The burner is of the type having three concentrically arranged ducts or passages in the burner body, the intermediate one of which is for fuel and the outer for the fluid under pressure for working the burner, whereby the ejector action of the burner when working draws air at atmospheric pressure into the central or axial duct and ejects the said air from the burner nozzle, and a regulating valve is provided for regulating the quantity of atmospheric air entering through the central passage. The bore of the central atmospheric air duct has formed in it right- and left-handed intersecting helical grooves or riflings. The burner body comprises as an integral casting outer and inner concentrically arranged tubular parts and has an adjustable conical nozzle member mounted on the open end of each tubular part. The aperture in the outer nozzle member is of double conical form, the face of the outer divergent cone being at an angle of 45° to the axis of the burner. H. E. T.

761. Burners with Vaporizers for Liquid Fuel. M. Coblitz. E.P. 344,321, 5.3.31. Appl., 27.6.30.

A description is given of a multiple burner with a vaporiser for liquid fuel. The vaporizer is approximately rhomboidal in cross section, is fitted with an internal electric heater, and extends lengthwise above two or more rows of burners. The latter are disposed transversely in the chamber, and are connected to each other by pre-heating air pipes and to the vaporizer by tubular legs extending downwards on both sides of the vaporizer. The burners in one row face gaps between those in another, the air feed pipes being outside the rows of burners. W. H. T.

762. Filtering Device for Liquids under High Pressure, Particularly Fuel Oil. F. V. Hangsted. E.P. 344,932, 16.3.31. Appl., 14.12.29.

The patent describes improvements in filters of the type in which filtering slits are established between the wall of a bore in a casing and a plug-shaped body fitted to this bore. Usually the two sets of channels are provided in the plug, but by placing one or both sets in the casing the machining costs are reduced and the possibility of damaging the edges of the filtering slits during cleaning is lessened. J. M. F.

Fluxes and Asphalts.

763. Emulsifying Action of Asphalt Fillers. A. R. Ebberts. *Roads and Roads Constr.*, 1931, **9**, 124–125.

The compressibility and resistance to water action of paving mixtures differ in an inexplicable manner, and the grain size, nature of surface, voids, etc., of various fillers have failed as dependable criteria. In view of the

powerful influence of inter-facial forces the emulsifying action of various dusts used as fillers was studied. By carrying out the experiments with a paving cement of 60 penetration at 210° F., it was shown that the results were comparable with those on an asphaltic oil used in the main experiments.

The oil, together with an equal volume of distilled water and known quantity of filler, was shaken in a bottle by hand. The emulsions produced fell into two classes, A (asphalt in water type) and B (water in asphalt type), the latter being blacker in appearance due to the continuous phase (asphalt) completely wetting the walls of the bottle. In all cases the dust was suspended in the continuous phase (not an invariable principle, however). Variations in the ratio of filler to asphaltic oil were examined.

Class A emulsions were produced by silica, iron oxide, gypsum and slag (?). Class B emulsions were produced by limestone, magnesium carbonate, hydrated lime, magnesium hydrate, portland cement and slag (?).

Therefore, a wet pavement in the presence of a group A filler will tend to disperse asphalt which will be lost in the drainage water. A group B filler is probably essential to the success of an asphalt pavement.

Among the peculiarities noticed were: calcium sulphate either as gypsum or "plaster of paris" gave incomplete emulsification, the sulphate ion probably exceeding the influence of the calcium ion. Slag dust also gave anomalous results.

Other experiments indicated that limestone is the best aggregate for asphaltic concrete in as much as limestone chips when added to group A emulsions caused them to separate into oil and water, the oil wetting the chips and the fillers remaining in the water. The efficiency of the breaking of the emulsion, therefore, depends upon the nature of the aggregate.

The superiority of limestone dust over silica dust in compressed pavement specimens was further evident when these were subjected to soft scrubbing action under running water, and also when the cylinders were put through a rubber-tyred abrasion test. The tests, therefore, indicate the advisability of using as high a percentage of calcareous materials in a paving mixture as is possible. C. I. K.

764. Improved Asphalt-Extractor. T. E. Stanton. *Eng. News Record*, 1931, **106** (38).

A new type of extractor is described in which the solvent is passed through an alundum filter stone 1 in. thick with the aid of 90 lb. air pressure. The maximum variation between the asphalt content used and extracted from eleven prepared samples containing a high percentage of 200v mesh material and asphaltic cement was 0·2%, the average variation being less than 0·03%. The solvent used is chloroform, approximately 85% of it being recovered. The cost of solvent per sample is 13c. The time of extraction with oil or asphaltic concrete mixtures is 15–30 mins. W. S. E. C.

765. Bitumen and Tar Emulsions. J. Leben and Ormul Products, Ltd. E.P. 344,562, 12.3.31. Appl., 18.12.29.

The patent deals with compositions adapted to be emulsified by the addition of a suitable proportion of water. They comprise bitumen, tar or other bituminous materials in association with acids of high molecular weight (fatty acids, and/or resin acids) an alkali metal silicate and less than 9% of water. The proportion of alkali present in the alkali metal silicate is in excess of that required to saturate the fatty or resin acid, the excess corresponding with one molecule for each 3 to 5 mols. of silicic acid present. W. H. T.

See also Abstracts Nos. 809, 810.

Special Products.

766. Catalytic Reduction of Carbon Monoxide at Ordinary Pressure, VII. Influence of Alkalis on an Iron-Copper Catalyst. S. Kodama and K. Fujimura. *J. Soc. Chem. Ind. Japan.* 1931, **34**. 14–163.

With a fine iron-copper catalyst (1 : 1) prepared from the nitrates, 140 litres of water-gas gave at 270°, with a space velocity of 230–240, 3·0 cc. of oil together with a small amount of solid resembling vaseline. The addition of sodium carbonate (0·005–0·05 mol.) to the catalyst increased its initial activity and favoured the production of higher hydrocarbons. This increase in activity was, however, not permanent, and the unactivated catalyst retained its activity for a longer time. The loss of activity of the alkaline catalyst seems to be due to the deposition of the higher hydrocarbons on its surface. The best result, 2·1 cc. of oil from 143 litres of gas, was obtained with a catalyst of the composition Fe : Cu : Na_2CO_3 = 1 : 1 : 0·005.

W. S. E. C.

767. Formation of Ethylene and Acetylene during Electric Discharges in Methane. K. Peters and O. H. Wagner. *Z. Physikal Chem.* 1931, **153**. 161–186.

The formation of acetylene and ethylene from mixtures of hydrogen and methane under the influence of the electric discharge has been studied under a variety of conditions of pressure, gas velocity and composition, energy input, and form of the reaction vessel. The minimum energy consumption for the production of acetylene is 13 kw.-hrs./m.³ and the most satisfactory value for the production of ethylene is 53 kw.-hrs./.m³ Under these conditions, and with a gas mixture containing 2 mol. of methane per mol. of hydrogen, the concentration of acetylene in the product is 4·2%, and of ethylene 0·8%.

It is apparently impossible to repress the formation of acetylene and increase the yield of ethylene, except at very low pressures, when the energy consumption becomes much greater than at 10–40 mm. The concentration of acetylene in the product increases with increase of the quantity of energy dissipated, the maximum attainable concentration being about 20%, whilst with ethylene the maximum does not exceed 3%. A study of the spectrum of the gas mixture during the discharge indicates that two reaction zones are present; in one only the ⋮CH bands are visible, whilst in the other the Swan spectrum, the Balmer series, and the carbon line spectrum also appear. The mechanism of the reactions is discussed. In the zone from which the Swan spectrum is emitted acetylene may be produced either by combination of two ⋮CH radicals or by the reaction of active hydrogen on the ·⋮C· radical. Ethylene is formed by the polymerisation of groups resulting from the decomposition of the methane molecule.

W. S. E. C.

768. Manufacture and Production of Wetting, Cleansing and Dispersing Agents. J. Y. Johnson. E.P. 343,098. 9.2.31. Appl., 7.8.29.

A process is described in which open chain or monocyclic aliphatic ketones containing at least 8 carbon atoms and/or their substitution products are sulphonated and compounded if desired with other wetting, cleansing or dispersion agents, protective colloids or salts or solvents of these agents.

W. S. E. C.

769. Purification of Carbon Black. J. P. Baxter and I.C.I., Ltd. E.P. 343,108. 10.2.31. Appl., 10.10.29.

Carbon black prepared by the action of chlorine on hydrocarbons is exposed for a short time while dispersed in a suitable gas to a temperature ranging from 150° C. to about 900° C. according to the nature of the gas employed. The carbon black is drawn from a hopper by an injector worked by the gas or gases used for purification and the mixture of carbon black and gas is passed upwards through a vertical heating zone at a velocity greater than the rate of settling of the particles of carbon in the still gas. W. S. E. C.

770. Oily Polymerisation Products from Butadiene or its Homologues. I. G. Farbenind. A.-G. E.P. 343,116, 16.2.31. Appl., 16.10.29.

The manufacture of oily polymerisation products from butadiene or its homologues is accomplished by exposing butadiene or the homologue in the form of vapour to a temperature of at least about 400° C. under ordinary pressure and if required in the presence of a catalyst. Butadiene passed over iron turnings maintained at 400°–500° C. gives an oil which boils chiefly over the range 110°–135° C. and consists mainly of ornylcyclohexenes. Similar results are obtained with aluminium, copper and nickel. Examples are also given of the method of working with isoprene and 1 : 3—dimethylbutadiene. W. D. S.

771. Treatment of Benzol obtained by Pyrolysis. D. A. Howes, W. S. G. P. Norris, S. T. Henderson and I.C.I., Ltd. E.P., 343,359. 19.2.31. Appl., 26.3.30.

The reactive products in benzol obtained by pyrolysis are mainly contained in the fraction boiling between 135° and 155° C. This fraction is therefore separated from the crude benzol and is hydrogenated either in the liquid or vapour phase and at atm. or higher pressures. A catalyst, especially such as are immune from sulphur poisoning, like molybdenum compounds, may be used. Saturated compounds including ethyl cyclohexane and the stable ethyl benzene are produced. The refining may then proceed separately or in bulk and for this purpose bauxite may be used or moderately concentrated sulphuric acid. E. N. H.

772. Process of Synthesising Organic Compounds containing Oxygen. Soc. Chim. de la Grande-Paroisse Ozote et Prod. Chim. E.P., 343,807, 26.2.31. Appl., 10.4.30.

Aliphatic organic compounds containing oxygen are produced by the action of CO on water in the liquid or vapour state. The process is carried out in the presence of artificial zeolites which result from the union of salts of a strongly alkaline character with metallates or salts of amphoteric metals. Neutral or appreciably neutral salts may be employed in addition. The artificial zeolites should be prepared in a medium with a P_H value between 8 and 6-8, preferably very close to 7 at 20° C. or at a corresponding P_H value if the temperature is different. After filtration and washing, the reduction of the zeolite is carried out on two phases, the first at a temperature of 200° C. under atmospheric pressure and dilute reducing gas, the second between the temperature of 300° and 500° C. under pressure and with concentrated reducing gas. Examples are given. W. D. S.

773. Pyrolysis of Hydrocarbons. I.C.I., Ltd. and D. Binnie. E.P. 343,881, 12.2.31. Appl., 12.11.29.

Hydrocarbons (methane) which are normally gaseous under atmospheric conditions, are converted into more unsaturated hydrocarbons (acetylene). A mixture of the gas with at least an equal volume of a gaseous or vaporised

water-soluble diluent (capable of ready removal from the reaction products (other than steam)) is subjected to a thermal treatment at temperatures above 1000° C. and at high space velocity of above 1000 reciprocal minutes.

Hydrochloric acid may be used as a diluent. W. H. T.

774. Treating Mineral Oils. K. T. Steik and H. A. Cassar, Assrs. to Standard Oil Dev. Co. U.S.P., 1,803,140, 28.4.31. Appl., 24.8.27.

This invention deals with the manufacture of colourless or water-white oils in which a refined distillate from crude oil is subjected to successive treatments with fuming sulphuric acid. The distillate of crude oil is agitated, after treatment with fuming sulphuric acid, for at least one hour with sulphuric acid of concentration not more than 90% and at a temperature between 70 and 120° C. The oil is finally purified by percolation through a bed of fuller's earth or contact with such clays or other absorption agents.

W. S. E. C.

Refining and Refinery Plant.

775. Refining of Light Petroleum Distillates. H. P. Rue and R. H. Espach. *Bur. Mines Bull.* 333. 1930. 111 pp.

Proper fractionation of the gasoline from a pressure distillate will decrease the amount of chemical treatment necessary to make the gasoline meet trade requirements. To obtain the maximum benefits of fractionation on the chemical treatment, a good degree of separation is necessary; there should be no overlaps in distillation range. In fractionation units a reflux (closed coil or open spray) at the top of the bubble tower is necessary to control the top temperature. When a closed-coil reflux is used, incoming charging stock may be preheated by pumping it through the coil. When an open-spray is used, the refluxing medium should be the material that is taken overhead as a vapour. The present methods of treating light distillates are described in detail. The influence of time, temperature and thoroughness of contact in treating with sulphuric acid is considered. The effects of acid treatment on carbon deposits, distillation ranges, gravity and colour are shown. The use of alkali is discussed. Studies with sulphur and sulphur compounds dissolved in gasoline are described and the effect of fractionation on colour and gum formation is discussed. W. S. E. C.

776. Flow Divider for Fractionation under Diminished Pressure. A. M. Schwartz and M. T. Bush. *Ind. Eng. Chem. Anal. Ed.*, 1931. **3** (2), 138.

The delivery line from a vacuum distillation apparatus is bent into a down pointing elbow and a reflux condenser is mounted on the still head returning all the vapour. The valve is sealed on to the elbow and consists of a U-tube, one end of which is sealed to the elbow and the other carries a valve. The U is filled with mercury and the rate of flow is regulated by raising or lowering the level of the mercury in the elbow. This is accomplished by means of the valve. This consists of a steel plunger which screws into a hollow metal cylinder. This cylinder is threaded internally to fit the plunger and externally on the top part to fit a packing cap. The whole is cemented into a taper at the end of the U-tube and is submerged in a well of mercury. W. L.

777. Unusual Features in New Refinery. R. C. Osterstrom. *Oil and Gas J.*, 1931, **29** (45). 61.

The recently constructed Toledo Refinery of the Pure Oil Co. contains three of the latest type Gyro vapour phase cracking units, compressor and absorption plant and treating equipment besides the usual distillation facilities, etc. Gasoline of high anti-knock value is obtained from Michigan crude by removing the first 40% of the gasoline fraction, which has a high anti-knock value and cracking the remainder in one of the Gyro towers, a yield of 60% of gasoline of ethyl standard being obtained. The gas oil fraction from Michigan crude is also cracked; the resulting benzine being blended with the straight run product from Mid-Continent crude, which is also worked up at this refinery.

The compactness of the refinery plant and the design of the fractionation tower permits of very economical running and flexibility. An interesting feature is that the complete plant was erected in five months, during the winter, including tankage for over half a million brls. Flow sheets and views of the plant are given. C. L. G.

778. Distillation of Tar. Barrett Co., Assrs., A. A. MacCubbin and J. Lavertnik. E.P., 318,897, 11.3.31. Appl., 11.9.29. Conv. (U.S.A.) 11-9-28.

The method is a continuous one in which the tar is first subjected to pipe-still distillation. It is heated to about 800° F. in the pipe-still coil and then discharged into a vapour separator at approximately atmospheric pressure. The separated vapours are withdrawn and condensed and the hot distillation residue is drawn off to a vacuum still while it is still at a high temperature and is subjected to a vacuum of not less than 29 in. of mercury by means of which practically all the constituents volatile at the temperature are vaporized.

The vapour separating chamber is situated below the vacuum still and the connection between the two includes a pressure reducing and regulating valve.

It is claimed that increased yields of distillate oils are obtained and that pitches with a m.pt. of at least 240° F. can be produced. W. L.

779. Steam Heated Heat-Exchangers. Schmidt'sche Heissdampf-Ges. E.P. 341,767, 22.1.31. Appl., 7.3.30. Conv. (Ger.), 25.4.29.

This invention consists of a steam-heated heat-exchanger having U-tubes arranged lengthwise in a casing. The legs of the U's are connected respectively to upper and lower headers.

The lower headers, through which the condensate is withdrawn, are rigidly connected to the casing, while the upper headers, through which steam is admitted, are connected to the casing by relatively long tubes having one or more bends in each to give flexibility.

These connecting tubes run longitudinally through that part of the casing that is traversed by the lower legs of the U-tubes. W. L.

780. Production of Sulphonic Acids. S. Pilat and J. Sereda. E.P. 343,530, 19.2.31. Appl., 19.9.29. Conv. (Pol.), 1.7.29.

Acid sludge obtained in the refining of oils is neutralised with lime and soluble calcium sulphonates separated by evaporating the filtered solution. The insoluble sulphonates in the precipitate are recovered by boiling with sodium carbonate and salting out the solution of sodium salts so obtained. T. A. S.

781. Distilling Hydrocarbon Oils. C. C. Miller, Assr. to Standard Oil Co. U.S.P. 1,786,357, 23.12.30. Appl., 15.6.27.

This invention deals with the design of a reducing valve and a modified form of expansion drum for use in a flash distillation apparatus. The valve comprises a tubular member provided internally with a conical valve seat and adapted to receive the hot oil from the pipe still.

A threaded stem, passing through a suitable packing nut and fitted externally with a hand wheel, carries a conical valve engaging with this seat. Beyond this actual valve portion the stem is provided with a short conical terminal which serves to maintain flow line conditions.

Beyond the valve seat the tubular member is provided with an outwardly flaring portion terminating in an open end. This flaring portion is provided at its throat with a number of lateral openings.

This valve may enter the expansion drum below the level of the oil contained in it.

If the valve is located above this oil level the tubular member is provided with a housing which completely encloses the lateral openings, and a pipe extends from this housing down into the body of oil in the drum. The open end of the flared portion is directed into an elbow pipe which extends downwards to within a short distance from the bottom of the drum.

W. L.

782. Distilling Oil. R. F. Gildehaus. U.S.P. 1,788,947, 13.1.31. Appl., 10.8.25.

A battery of stills is connected in series so that oil is free to flow by gravity through them. Each still has a special heat absorbing section composed of two drums below the main shell. These are connected to the shell by vertical ducts and are interconnected by a bank of inclined tubes. Each still is fitted with a pump to maintain positive directional circulation of the oil through the still and heating section. Each still has an independent condensing system and vacuum may be applied to the whole system. W. L.

783. Pipe Still. G. W. Watts, Assr. to Standard Oil Co. U.S.P. 1,789,267, 13.1.31. Appl., 18.7.25.

In this still the return bends in general use are replaced by headers supporting the ends of the pipes. Certain of these are separated by supporting feet which in turn are shielded from flue gases or heat by the brickwork. By the use of these headers steel or cast iron supporting tube sheets are eliminated.

Plugged holes are provided opposite the tube holes in the headers, so that the tubes can be cleaned and rolled or new tubes inserted.

The general design of the still is such that it is possible to inspect practically the entire surface of every tube. A counter flow is obtained except for a two-layer pipe screen in the pass directly above the stoker. This screen circulates comparatively cold oil and serves to protect the top arch from destructive overheating. W. L.

784. Dehydrating and Distilling Heavy Hydrocarbon Oils to Recover Constituents therefrom in Liquid Form. A. S. Knowles, Assr. to Tar and Petroleum Process Co. U.S.P. 1,789,312, 20.1.31. Appl., 13.12.26.

This plant comprises a furnace designed to provide a horizontally elongated heating chamber adapted to receive a cylindrical basket or holder, which is partially filled with a loose porous bed of carbonaceous material and is capable of being rotated so as to preserve its porosity. Connections are provided for introducing pre-heated gas or steam under pressure to the body of the bed.

Distillates pass out of the basket through openings in the periphery and are discharged from the heating chamber through suitable ducts.

In operation the basket is turned so that one perforated section is at the top and the other is at the bottom. Charging oil is fed to the top of the porous bed, and preheated gas or steam under pressure is admitted to the body of the bed. Volatile hydrocarbons pass out through the top of the basket, while the residual liquid collects in gutters at the bottom and is conducted to a transverse trough and thence to a receiver. At suitable intervals the feed is stopped, and the basket rotated through a half turn or a turn to maintain the porosity of the bed. W. L.

785. Distillation Control. W. K. Lewis, Assr. to Standard Oil Development Co. U.S.P. 1,789,339, 20.1.31. Appl., 31.3.28.

A small proportion of the bottoms from a distillation is continuously withdrawn at a uniform rate, and is heated under conditions of uniform heat input to produce vapour and an unvaporised residue. The vapour produced is carefully rectified, and the temperature of this rectified vapour is measured. The conditions of the large distillation are modified to the extent necessary to hold this temperature close to a pre-determined point. W. L.

786. Distilling Oils. J. R. Schonberg, Assr. to Standard Oil Dev. Co. U.S.P. 1,791,937, 10.2.31. Appl., 28.5.25.

This distillation apparatus is composed of two drums situated one above the other. Heating flues pass longitudinally through each drum. Combustion gases pass directly into the flues in the lower drum prior to any substantial contact with the outside shells. From here they pass through the flues in the upper drum and finally over the outside shell of the upper drum.

Oil is charged to the upper drum and overflows from here to the lower down an overflow pipe. Each drum is fitted with vapour pipes and residue run-off connections.

The still may be used for vacuum distillation, and relatively small drums can be used, thus doing away with the internal reinforcing which is usually essential in large shells. W. L.

787. Petroleum Treating System. S. J. Dickey and E. W. Roth, Assr. to General Petroleum Corpn. U.S.P. 1,792,003, 10.2.31. Appl., 15.11.26.

The liquid is purified by counter-current washing with a mixture of refined product and reagent or reagent sludge. The process is carried out in a series of washers, the most nearly refined oil being treated with fresh reagent in the last washer. Sludge from this washer is withdrawn, remixed with a proportion of refined oil and pumped to the last but one washer, and so on through the series so that the raw oil is treated with the nearly spent reagent. W. L.

788. Operating Oil Stills. B. Broido, Assr., Superheater Co. U.S.P. 1,794,439, 3.3.31. Appl., 31.5.23.

This invention relates to stills in which viscous oil is heated while being forced through the apparatus.

The oil is first passed through a number of small coils exposed to the most intense heat of the furnace. Here the viscosity is rapidly reduced to a point where the oil flows freely and requires relatively little force to move it through the still.

After passing through these coils any vapour is removed and the oil passed through the furnace proper under the normal counter-current conditions.

The vapours which are removed after the initial heating are added to the liquid at the end of the second heating. W. L.

789. Treating Petroleum. W. F. Bleecker. U.S.P. 1,794,668, 3.3.31. Appl., 1.5.28.

This process consists in mixing under pressure an oxidising gas with petroleum containing corrosive sulphur compounds. Nascent gas is preferable as its action is quicker and more positive.

Brine is electrolysed in the presence of petroleum. The chlorine produced is passed through the petroleum, but the hydrogen generated at the cathode is removed without allowing it to come into contact with the oil. W. L.

790. Refining Hydrocarbons. F. A. Apgar, Assr. to Sinclair Refining Co. U.S.P. 1,795,067, 3.3.31. Appl., 14.9.28.

Cracked spirit is refined by passing it through an adsorptive catalyst in the vapour phase. The raw vapour mixture from the cracking operation is scrubbed for the separation of unvaporised oil and tarry matter. An oil condensate of higher b. pt. than the gasoline is then separated and passed as a liquid washing agent through the catalyst together with the gasoline. The washing agent and polymers are separated from the refined vapour mixture and passed directly into the scrubbing operation. W. H. T.

791. Distillation of Oil. J. E. Bell, Assr., Sinclair Refining Co. U.S.P. 1,795,070, 3.3.31. Appl., 1.11.24.

Vapours from the still pass up a reflux tower and from the top of this the vapours pass to the tubes of a heat exchanger. The cooling medium in this heat exchanger is fresh oil. As this overflows from the heat exchanger it passes to the reflux tower which it enters at an intermediate point. Any part of the fresh oil that is vaporised in the heat exchanger is introduced to the reflux tower at a somewhat higher intermediate point and through separate connections. A lighter oil, corresponding to the desired distillate is introduced into the top of the reflux tower. Residue is continuously withdrawn from the still to maintain the desired oil level.

Vapours from the bottom of the heat exchanger are condensed in the usual way. It is claimed that close fractionation can be obtained with improved heat economy. W. L.

See also Abstract No. 801.

Cracking.

792. New Corrosive Proof Pressure Vessels. O. E. Andrews. *Oil and Gas J.*, 1931, **29** (45), 134.

Methods of combating corrosion in cracking plant chambers have been extensively investigated by the A. O. Smith Corporation. A successful method, though too costly for general application, is the coating of the internal surface with an alloy weld. A commercially practicable method was then developed for the bonding of a corrosion-proof alloy as a lining of the entire vessel. Twelve-gauge chrome nickel iron of the 18–8 class has been used with success, the bond of the alloy to the steel developing a shearing strength of 8000 lbs. The bond practically makes the alloy an integral part of the vessel, so that

temperature stresses do not arise to any appreciable extent. Parts of Smith welded stills protected with this type of alloy show no signs of chemical or physical deterioration after several years in operation. Although the cost of the alloy lining is high, economies are effected by the reduction in cost of replacement and repairs, shut down costs for such replacements and repairs, loss in efficiency due to decreasing wall thickness, and hazard costs incidental with corroding equipment. Coating with non-metallic lining is one of the most satisfactory and economical solutions up to date, although the coating is susceptible to injury, while the heat insulating effects make cleaning and inspecting longer, more expensive and unpleasant. C. L. G.

793. Transforming Hydrocarbons Having a High Boiling Point into Volatile Hydrocarbons. F. P. E. S. Duplan. E.P. 339,841, 18.12.30. Appl., 20.1.30. Conv. (Ger.), 21.1.29.

The oils are heated in the liquid phase, at a pressure considerably above their vapour pressure, in a tube in which they are caused to circulate at a definite velocity. For a tube up to 10 mm. diam. this velocity lies between 6 and 72 cm./sec. The oils are maintained at a temperature of 360° C. for between 1 and 1½ min. The operation is carried out in a furnace composed of a series of straight parallel tubes through which the oil is passed in succession. These tubes are embedded in a block of conducting material which is infusible at the temperature of operation. W. L.

794. Cracking Oils. A. E. White, Assr. to Bethlehem Steel Co. E.P. 345,005, 19.3.31. Appl., 18.1.30.

In the process described the reflux from the dephlegmator is not returned to the still, as it is claimed that owing to its low b. pt. as compared with that of the charging stock very little, if any, further cracking takes place under the conditions obtaining in the still, and waste of fuel is thereby avoided.

The still consists of a vertical cylindrical vessel whose lower part is protected from direct furnace heat by an apron wall.

The preheated charging stock, gas oil, is heated in this still to about 600° C. and under a pressure of 7–8 atm. The vapours pass counter-current to the charging stock through the dephlegmator of rectangular shape and divided vertically into four compartments. Four cuts of reflux are thus obtained and these are united into two main fractions.

It is recommended that the heavier of these be used for works fuel and the lighter for reducing the gravity and improving the viscosity of heavy fuel oils.

The condenser is of the same type as the dephlegmator and a heavy condensate similar to the lighter one from the dephlegmator can be obtained and used with this latter. Where a number of stills are employed the heavier reflux fractions can be treated in a secondary still under suitable cracking conditions. H. B. T.

795. Treatment of Oil. D. G. Brandt, Assr., Doherty Research Co. U.S.P. 1,784,087, 9.12.30. Appl., 31.1.22.

The charging stock is first preheated to about 450° F. in vapour heat exchangers. It then passes down through a dephlegmator maintained under a pressure of about 100 lb., where it is scrubbed and the lower boiling constituents removed by contact with an ascending current of hot vapours. The scrubbed oil and the condensate from the scrubbing vapours then pass into the cracking chamber which is maintained under the same pressure as the dephlegmator. As it passes down through this chamber a portion is cracked and a portion vaporised and the liberated vapours pass up through the dephlegmator.

The residual oil from the bottom of the chamber passes into the lower drum of a cracking still and thence through a series of small tubes to a vapour separating drum. These tubes are situated in a combustion chamber and are in direct contact with the hot combustion gases. The circulation in the tubes is comparatively fast to prevent the deposition of carbon, and though the oil is raised to a cracking temperature very little cracking takes place, but a large amount of oil is vaporised. These vapours are separated from the oil in the upper drum.

The oil from this drum passes through a separator provided with a pocket to collect carbon and heavy residues and is introduced into the upper portion of the cracking chamber. The vapours from the drum pass directly into the lower portion of the cracking chamber and a series of baffles and trays cause them to take a circuitous path through the descending oil. Carbon and tarry matter is deposited from the oil as cracking proceeds, and working its way to the bottom of the chamber accumulates in a pocket there and may be drawn off at will. The exit from cracking chamber to still is positioned some distance above this pocket to avoid the circulation of carbon, etc.

Practically all the cracking takes place within the heat-insulated cracking chamber, so that there is little or no deposition of carbon on the heat-transferring surfaces in the still. W. L.

796. Oil Cracking. J. H. Welch. U.S.P. 1,784,562, 9.12.30. Appl., 6.7.27.

This process takes into consideration not only the pressure and temperature conditions, but also the time factor during which the cracking reaction takes place.

The charging stock is preheated by scrubbing with the cracked vapours from the cracking chamber, and then passes through a pipe still where it is heated to a cracking temperature. It is then fed into the top of a heavily-insulated reaction chamber. The liberated vapours pass up to a reflux tank situated above the reaction chamber and are here scrubbed by direct contact with the incoming charging stock. Above this oil they pass through conduits in a funnel-shaped wall, where a swirling motion is imparted to them, and then to the bottom of a bubble tower where they are fractionated. The lower portion of this tower is kept full of condensate and the incoming vapours are scrubbed as they rise through the tower. This condensate is withdrawn and discharged to storage as distillate. The residuum from the bottom of the reaction chamber is withdrawn and used as a heating medium to maintain the liquid in the bottom of the fractionating tower at its desired temperature. It then passes through a carbon trap, where it is cooled by contact with cold charging stock and finally is pumped to fuel storage. W. L.

797. Pressure Still for Cracking Oils. D. A. Young, Assr., Sinclair Refining Co. U.S.P. 1,788,006, 6.1.31. Appl., 15.7.27.

This relates to the arrangement of the regulating valve in the discharge line of pressure cracking stills. It is claimed that it improves the regularity of operation of such lines and also assists in keeping them clean over indefinite periods of time.

The valve is arranged within the still and at the extreme inner end of the discharge line. The reduction in pressure which takes place at this valve is made effective as soon as the oil enters the discharge line so that the increase in velocity due to vaporisation resulting from this reduction in pressure is also made effective throughout the discharge line. This inhibits the formation of deposit. W. L.

798. Tube Still for Oil Distillation and Cracking. H. Thomas, Assr., Sun Oil Co. U.S.P. 1,788,213, 6.1.31. Appl., 14.6.27.

This still is composed of an upright gas-combining and heating chamber with a bank of tubes adapted to convey oil in the upper parts.

A series of laterally disposed Dutch ovens communicate with the lower part of the chamber below the tubes. At the forward ends of these ovens are air inlets, and cooled gases from the region of the tubes are recirculated to the region near the junction of the floor of the Dutch ovens and the lower front part of the upright chamber by means of channels and power-driven fans.

From these channels is a branch channel communicating with a stack adapted to provide draft sufficient to pull a proportion of the cooled gases against the pull of the power fans.

Below the tubes is an unobstructed space in the upright chamber where the combustion gases are diluted with recirculated gases to produce a mild and even temperature under and around the tubes.

A fresh-air conduit is situated in heat exchange relation with one of the recirculating channels and delivers warm air to the air inlet of the Dutch ovens. W. L.

799. Distilling Hydrocarbons. E. Piron. U.S.P. 1,794,542, 3.3.31. Appl., 20.1.27.

Metal blocks in a series in the form of a chain or conveyor are heated to suitable temperatures and passed through a distillation zone where hydrocarbons, either solid or liquid, are applied in thin layers and distilled. The heat stored in the blocks distils the hydrocarbon and forms a residue or coke which is later removed and the volatile products formed are recovered and used.

The blocks are removed from the distillation zone before the volatile constituents have been permitted to become overheated by contact with the hot surfaces for an appreciable length of time, thus obviating over-cracking.

It is desirable for the coke to have a volatile content of not more than 20%, and to attain this the temperature of the blocks ranges from 1050° to 1350° F., and the vapour temperature of the distillate from 500° to 1000° F. when distilling petroleum oil. Under these conditions the blocks can be passed through the combustion chamber at a sufficiently rapid rate to prevent their surfaces reaching such high temperatures after distillation has taken place as to overcrack the vapours to an undesirable extent. W. L.

800. Removing Carbon Deposit from Metal Surfaces. C. B. Watson, Assr., Pure Oil Co. U.S.P. 1,796,878, 17.3.31. Appl., 27.7.27.

The internal surfaces of tubes used in oil heating furnaces are cleaned by passing a spraybead longitudinally through them. Water is forced radially from the spraybead and impinges on the coke deposit, the pressure being of sufficient magnitude to effect the mechanical removal of the adhering deposit from the walls of the tubes. W. L.

Chemistry of Petroleum.

801. Chemistry of the Doctor Sweetening Process. A. Lachman. *Ind. Eng. Chem.*, 1931, **23**, 354–357.

The sweetening of petroleum distillates by doctor solution is now recognised to be a complicated chemical process in which several actions take place simultaneously. The litharge in the treating solution becomes converted into

lead sulphide which itself is capable of acting catalytically in a variety of ways. It is also capable of adsorbing mercaptans from the solution. If air agitation is employed during the sweetening process the lead sulphide is wholly or in part reconverted into sodium plumbite, while the mercaptans dissolved in the alkali present are converted into disulphide by the air. This oxidation of the mercaptans takes place both in the aqueous phase and at the interphase, so that it is the mercaptan ion and not the mercaptan itself which undergoes oxidation. Furthermore, the rate of oxidation is governed by the rate of diffusion of the oxygen into the alkaline film. Powdered sulphur acts as a catalyst in promoting this oxidation but this effect is probably because it is a solid and not on account of its chemical behaviour. The normal operation of sweetening by plumbite yields sodium sulphide and this also is altered by oxidation being converted into sodium thiosulphate. The sodium sulphide is only detrimental in that it competes with the mercaptan for the oxygen. If present in small amounts only it makes little difference to the rate of mercaptan oxidation, but if present in larger amounts larger quantities of air and sulphur are required for sweetening. This explains why when sweetening distillates containing the more refractory mercaptans a larger quantity of sulphur is required and the aerial agitation must be continued longer. It is suggested on the basis of these results that it is advantageous to remove hydrogen sulphide before the sweetening treatment and also to add the whole of the sulphur required, calculated on the mercaptan content, in a very finely divided condition, at once. Excess of sulphur, if it were not for the corrosive action of the residual sulphur, would actually be desirable.

S. F. B.

802. Some Copper Mercaptides and Their Reaction with Carbon Disulphide. W. E. Duncan, E. Ott and E. E. Reid. *Ind. Eng. Chem.*, 1931, **23**, 381–384.

The present study was undertaken to obtain a more exact knowledge of the copper mercaptides. The complete series of copper mercaptides from methyl to *n*-nonyl and from *sec*-propyl to *sec*-nonyl can be prepared by the interaction of the mercaptan in benzene solution with cupric acetate in aqueous solution. The mercaptide obtained is the cuprous compound, while some of the mercaptan is converted into the corresponding disulphide in all cases. Typical mercaptides have been analysed and their properties noted. The cuprous mercaptides are insoluble in water, alcohol, ether and benzene, with the exception of the *sec*-amyl, *sec*-hexyl, *sec*-heptyl, *sec*-octyl and *sec*-nonyl mercaptides, which are soluble in benzene and ether. They are at first light yellow in colour, but show decomposition analogous to that observed with lead mercaptides, slowly becoming deeper in colour until they eventually turn deep brown. They also react with elementary sulphur in a way similar to that observed for the lead salts. Cuprous mercaptides readily react with carbon disulphide to form red brown alkyl cuprous trithiocarbonates, or Cu—S—CS—S—R, which are more soluble in organic solvents than the corresponding cuprous mercaptides. Unlike lead, copper does not appear to form basic compounds and the mercaptides show less tendency to hydrolyse.

Copper mercaptides are formed by the action of mercaptans in benzene solution on metallic copper, copper sulphide, copper oxide and by interaction with butyl copper phthalate. The formation of copper compounds soluble in benzene, reported by Slagle and Reid, when measuring the "adsorption" of mercaptans by copper sulphide is thus readily explained.

S. F. B.

803. Synthesis from Natural-Gas Hydrocarbons. I. Caproic Acid from Pentane. H. B. Hass and J. R. Marshal. *Ind. Eng. Chem.*, 1931, **23**, 352–353.

Conditions have been worked out for obtaining good yields of alkyl cyanides from the corresponding chlorides by means of sodium cyanide. Yields of 70%, with a recovery of 25·28% of unchanged chloride have been obtained with the *n*-amyl chloride. Complete separation of the chloride, b. pt. 105–107° C., from the nitrile, b. pt. 161–162° C., was accomplished through the constant-boiling mixture which is formed with alcohol and which boils at roughly 72·5° C. The use of sodium iodide, which acts as an intermediate compound catalyst, increased the yield to 90%. Other normal chlorides also gave good yields, but secondary chloride and bromides gave poor yields (roughly 30%). No nitrile at all could be obtained under these conditions from the tertiary amyl chloride or bromide. The hydrolysis of the *n*-amyl cyanide (capronitrile) was carried out by means of 67% sulphuric acid. The reaction is exothermic and once started by warming under reflux completes itself in 30 min. Pure caproic acid is recovered by distillation after water has been added to prevent separation of ammonium bisulphate. The final yield is determined by the purity of the amyl chloride employed. It is interesting to note that amyl and butyl chlorides resemble carbon tetrachloride ethyl, iodide, benzene, heptane, etc., in forming binary minimum boiling azeotropic mixtures with ethanol and ternary azeotropic mixtures with ethanol and water. In the conversion of the *n*-amyl chloride to the corresponding nitrile methanol or *iso*-propanol can be used instead of ethanol, but if methanol is used it must be removed by distillation and replaced by either ethanol or *iso*-propanol before fractionation, as it does not form an azeotropic mixture. S. F. B.

804. Partial Oxidation of Methane in the Presence of Oxides of Nitrogen. D. F. Smith and R. T. Milner. *Ind. Eng. Chem.*, 1931, **23**, 357–360.

The partial oxidation of methane is of considerable interest in connection with the chemical utilisation of natural gas. It was claimed by Bibb (U.S.P. 1,392,866 (1922); re-issue 15,789 (1924)) that the presence of small amounts of nitric acid or its decomposition products considerably increase the quantity of methane oxidised to formaldehyde and this effect has now been further examined. Complete data on the reaction in small glass apparatus have been obtained under varying conditions of temperature, heating time, gas composition and amount of catalyst using nitric acid vapour, nitrogen dioxide and nitric oxide. Under the conditions studied, the production of formaldehyde by the partial oxidation of methane in the presence of oxides of nitrogen would not be commercially feasible. Suggestions are made as to the possible mechanism of the reaction. S. F. B.

805. Oxidation of Methanol with Air over Iron, Molybdenum and Iron-Molybdenum Oxides. H. Adkins and W. R. Peterson. *J.A.C.S.*, 1931, **53**, 1512–1520.

The oxidation of methanol with air over various metal and oxide catalysts at 300°–700° C. results in the formation of varying amounts of formaldehyde carbon monoxide and carbon dioxide. The characteristics of molybdenum oxide, iron oxide and a mixed iron-molybdenum oxide as displayed in the catalysis of the oxidation have been described. The molybdenum oxide was 100% efficient for the oxidation of methanol to formaldehyde, since this is the only reaction which occurred. Only about 38% of the methanol could be converted to formaldehyde. Iron oxide was a very active catalyst but

it induced the formation of carbon dioxide almost exclusively. A catalyst containing equal atomic amounts of iron and molybdenum converted over 90% of the methanol passed over it into formaldehyde, the other product being carbon monoxide. It is thus by far the best catalyst so far described, for the oxidation of methanol. The catalyst was quite stable and may be used continually for months.

In the case of molybdenum oxide there is evidence that the reaction between methanol and oxygen takes place when both reactants are on the surface of the catalyst.

Evidence is presented which indicates that the catalyst surface of these oxide catalysts is continually renewed during use. Therefore the relationship between an effective catalyst and the reactants must not only be such that the former brings about the reaction of the latter, but the reactants must maintain the catalyst in the state of oxidation which is favourable to the desired reaction. W. D. S.

806. Nuclear Syntheses in the Olefine Series. II. 1, 4-Diolefines. B. H. Shoemaker and C. E. Boord. *J.A.C.S.*, 1931, **53**, 1505–1512.

The nuclear syntheses of unsaturated hydrocarbons has been extended to the syntheses of 1, 4-diolefines. This extension has been made possible through the preparation of allyl magnesium bromide (Gilman and McGlumphy, *Bull. soc. chim.*, 1928 (4) **43**, 1322). Addition of $\alpha\beta$-dibromo-alkyl ethyl ether to a slight excess of allyl magnesium bromide leads to the formation of an α-allyl-β-bromo-alkyl ethyl ether, compounds of which type are readily steam distilled. The conversion of the β-bromo ethers into the corresponding unsaturated compounds has also been improved by replacing the ethyl alcohol used in the zinc reduction by *n*-propyl or *n*-butyl alcohol. 1, 4-Pentadiene, 1, 4-hexadiene and 1, 4-heptadiene and the corresponding α-allyl-β-bromo-alkyl ethers are described.

The rule that unsaturated compounds with a double bond in the alpha position boil lower than the corresponding saturated hydrocarbons has been confirmed. It has been further shown that in any given family the continuous chain diolefines form a definite series with gradually increasing boiling points depending upon the relative positions of the double bonds.

The 1, 2, 4, 5-tetrabromo paraffins corresponding to the diolefines described, have been prepared and found to show evidences of isomerism analogous to other polybromo aliphatic derivatives (cp. polybromo fatty acids). W. D. S.

806A.—Ultrafiltration of Petroleum. A. Zaharia and E. Lucatu. *Bul. Soc. Chim. Romdnia.* 1930. **12**. 90-4.

In an attempt to settle the question as to whether paraffin, resins and asphalt are present in petroleum in colloidal or in true solution, petroleums of different types from five localities were subjected to ultrafiltration. Vulcanised rubber membranes of 0·04 mm. thickness and a vulcanisation coefficient of 1·7 were used in the Brukner apparatus under a pressure of 150 atm. Two membranes were used that gave an effective filtration surface of 25 sq. cm. The resins of petroleum which are separable by 70% ethyl alcohol were found to be ultrafilterable. All the paraffin in the petroleums used was ultrafilterable but the asphalt, both hard and soft, was removed quantitatively by ultrafiltration. This behaviour indicates that the asphalt is colloidal but that the resins and paraffins are not. The asphalt retained by the ultrafilter was dissolved in benzol and cyclohexane and was again retained by the filter. Several passages of oil through the same filter removed the resins and left the pure dry asphalt, which dissolved readily in that portion of the petroleum that was ultrafiltered. This experiment shows that the asphalt exists in petroleum as a lyophile colloid. W. S. E. C.

Analysis and Testing.

807. Fluorescence in Mineral Oils. K. Trautzl. *Erdöl und Teer*, 1931. 7, 193.

The author disagrees with the conclusions of Tausz and Rabl (cf. Abs. 511), that the deblooming of mineral oils by the addition of aromatic nitro-compounds is a chemical process depending on the formation of addition compounds by means of residual valencies.

Experiments were carried out on the fluorescence of oils in the light of the quartz lamp and on the effect of the addition of aromatic nitro-compounds. If nitro-glycerine is dissolved in a solvent, such as alcohol or ether, which itself does not fluoresce, and exposed to the light of a quartz lamp, a fluorescence is observed. If a small amount of, say, nitrotoluol or nitro-naphthalene be added, the fluorescence is completely extinguished. Nitro-glycerine is chemically a very simple body, at the most contaminated with the dinitro-compound, and no fluorescence carriers can be present. Hence, even if one assumes residual valencies for the tri-nitrotoluol, no addition compound can be formed with nitroglycerine. It is hence concluded that the phenomenon is optical and not chemical, and that similar considerations apply in the case of oils which fluoresce in daylight.

These properties are useful in determining whether a nitroglycerine explosive contains also aromatic nitro-bodies, since fluorescence in the light of the quartz lamp indicates the absence, and non-fluorescence the presence, of these bodies.

E. B. E.

808. Considerations on Absolute Viscometers. Louis. *Ann des Comb. Liquides*, 1930, **5**, 949–958.

The author first points out the divergent results obtained with the Engler viscometer and the still greater errors occurring when these results are converted to absolute units. The advantages of employing absolute viscosities are discussed and for commercial use the Ostwald relative viscometer calibrated with a series of suitable liquids whose viscosities have been determined in a true absolute viscometer, is recommended. The upward flow type is more suitable as drainage errors are reduced.

An absolute viscometer suitable for use at temperatures higher than atmospheric is described. This is a modification of the viscometer of Vlès, with the capillary tube vertical and enclosed in a thermostatic bath. The following advantages are claimed for the instrument: accurately constant pressure, accurate temperature control over a wide range, ease of assembly, directness and ease of measurements and an accuracy of within 1%.

E. B. E.

809. Surface Tension of Asphaltic Bitumens and Allied Substances at Various Temperatures. F. J. Nellensteyn. *Roads and Road Constr.*, 1931, **9**, 127–128.

Of the various methods available for surface tension measurement, the capillary rise method was useful between temperatures of 180–200° for asphaltic bitumens, but the best method found was that of the air-bubble pressure (using nitrogen to avoid oxidation), after the procedure given by Nellensteyn and Roadenburg (*Kolloid-chemische Beihefte*, 1930, **31**, 434).

The substances used were [the properties are given in the order, penetration, melting point (ball and ring), insol in CS_2, insol. in ether]: (1) oxidised Venezuelan asphaltic bitumen: 74, 64·2° C., traces, 22·7%; (2) Spramex (Royal Dutch Shell): 194, 42° C., traces, 19·6%; (3) Shelfalt (R. D. S.), 50–60; 55·7° C., traces, 22·7%; (4) Shelfalt (R. D. S.), 40–50; 44, 58·5° C., traces, 22·0%; (5) asphaltic bitumen from Trinidad épuré; (6) coal-tar pitch of Dutch origin, melting point 60·2° C., free carbon 10·7%.

Surface tension-temperature data were obtained over a wide range of temperature for the asphaltic bitumens and for the oily constituents (in which the asphaltic bitumen micelles are dissolved) which were prepared from the bitumens. In both sets of curves a deflection point was a characteristic. The conclusion arrived at is that when heating a pitch or asphaltic bitumen, a sudden decrease in the cohesion of the colloidal liquid often occurs, and above this temperature the substance behaves like a lubricating oil. A temperature above this point is necessary for the mixing process (usually 30–40° C. above this point) above which temperature the surface tension is a linear function of the temperature.

The deflection points for the six substances were: (1) 150° C.; (2) 117° C.; (3) 145° C.; (4) 138° C.; (5) 132° C.; (6) 80° C. Hence a lower penetration does not always correspond with a higher deflection point, even with similar bitumens [vide (3) and (4)].

The highest mixing temperature is clearly required by blown asphaltic bitumen and coal-tar pitch shows higher values for surface tension than asphaltic bitumens of about the same melting point.

The work is incomplete and it is to be extended to natural asphalts.

C. I. K.

810. Methods of Determination of the Paraffin Content of Asphalts. W. v. Piotrowski and H. Burstin, *Petr. Zeit.*, 1931, **27**, *Asphalt und Strassenbau* (3), 3–7.

Since asphalt for road construction must have a definite paraffin content the determination of this property is of great importance.

A review of the work of the mineral oil section of the Polish Standardisation Committee is given and the paraffin determination according to the methods of Engler-Holde, Marcusson and Schwarz described in detail. The paraffin contents of (1) asphalt obtained from paraffin-free petroleum, (2) a blown petroleum asphalt obtained by oxidation of cracked residues by means of air, and (3) an asphalt produced by concentrating paraffinic petroleum were determined by the three named methods and the results tabulated; Engler-Holde's method was adopted as the most suitable standard method for the determination of paraffin in asphalt.

W. S. E. C.

811. Effect of Light on the Determination of Ethylene. J. L. Oberseider and J. H. Boyd, Jr. *J. Ind. Eng. Chem., Anal. Edn.*, 1931, **3**, 123.

The determination of ethylene when carried out in sunlight can be made more accurate when the Williams pipette is painted black, except for a narrow vertical strip. The activity of bromine upon heavy hydrocarbons is thereby increased so that the ethylene results obtained by absorption in aqueous bromine are otherwise high when made in sunlight.

W. S. E. C.

812. Dimensional Analysis Applied to the Thermal Conductivity of Liquids. J. F. Downie-Smith. *Ind. Eng. Chem.*, 1931, **23**, 416.

If the assumption is made that the thermal conductivity of a liquid is a function of molecular weight, density, specific heat, viscosity, gas constant,

thermal expansion and compressibility, the following equation may be produced—

$$\frac{k\,K^{1/3}\,\lambda^{1/3}\,Z^{1/4}}{c^{9/3}\,\rho^{1/3}\,m^{5/12}} = \phi\left(\frac{m\,c}{a}\right)$$

where k = thermal conductivity, K = compressibility per unit volume, λ = thermal expansion per unit volume, Z = viscosity, c = specific heat per unit mass, ρ = specific gravity, m = molecular weight, a = gas constant.

This equation is modified and a graph is given connecting

$$\frac{k}{\sqrt[3]{\rho c}} \text{ and } \frac{c}{K^3\,\lambda^{1/3}\,m^{1/3}\,Z}$$

P. D.

813. Apparatus and Methods for Precise Fractional Distillation Analysis. W. J. Podbielniak. *Ind. Eng. Chem., Anal. Ed.*, 1931, 3, 177–188.

The history of the development of the Podbielniak apparatus for precise fractional-distillation analysis is outlined, including a complete survey of low-temperature fractionation methods described in the literature. A comprehensive description is given of the apparatus and analytical procedures in their present state of development, including such topics as accuracy limitations, checking methods, sampling and interpretation of results. Precision-type fractionating columns are described for the first time. They have increased fractionating efficiency and greater sensitivity over the regular-type column. Diagrams are given of two different constructions and their action is explained. More efficient fractionation is made possible (especially in the case of small samples) because of less hold-up of vapour and liquid, and because of the somewhat greater length of packing possible for the same over-all length. The total volumetric capacity can be reduced to less than 6 c.c., by filling the distilling bulb of either precision column with mercury, thus greatly increasing the sensitivity to very small samples. Moreover, owing to the more efficient reflux arrangement, the consumption of liquid air using a precision-type column is materially less than with the regular column. Separation of highly complex mixtures of saturated and unsaturated hydrocarbons into their individual components with high accuracy, and in a short time may be accomplished with this new type of column.

Numerous fractional-distillation curves and check comparisons are given to show the performance of the apparatus on various kinds of gaseous and liquid mixtures. The apparatus and method are widely used in the manufactured-gas, natural-gas and petroleum industries, and are presented by the author as an accurate analytical tool for the complete analysis of gaseous (or volatile liquid) mixtures of all kinds, which are too complex for the conventional tests. W. D. S.

814. Refractometric Dispersion of Mineral Oils. E. Vellinger and N. Orlovski. Ann. des Comb. Liquides, 1930, 5, 959–976.

The dispersion of the refractive index for different wave-lengths of light has been used to some extent for the examination of spirits. The authors have extended this work to heavier oils.

Various oils were distilled in the cathode-ray vacuum, and the refractive indices of the fractions measured for three wave-length bands (578, 546 and 435 approx.) of the mercury vapour-lamp. These results and also Gladstone's constant $\left(\frac{n-1}{d}\right)$, the density, and $\frac{\Delta n}{d}$ are given in tables and curves for twelve oils (mainly transformer) of different degrees of refining.

The more highly refined the oil, the lower is the refractive index of corresponding fractions and the less is the dispersion, considering oils from the same crude. The density is also lower. If the refractive index is plotted against the density of the fraction, a more or less smooth curve (verifying Gladstone's formula) is observed for some oils. Irregularities in the curve indicate the presence of hydrocarbons of series different from that of the main constituents. E. B. E.

814a. Photolysis of Mineral Oils. E. Vellinger and P. Marchand, *Ann. des Comb. Liquides*, 1930, **5**, 977–986.

The effect of varying conditions on the alteration of dilute solutions of mineral oils when exposed to light has been studied. Both daylight and the light from a mercury-vapour lamp were employed, the development of yellow colourations being followed by means of a Koenig-Martens spectrophotometer.

In the absence of air—*i.e.*, in atmospheres of nitrogen or carbon dioxide—the solutions in general become yellow. If air was present a very fine precipitate, observable by the Tyndall effect, was produced. This is probably due in the first case to polymerisation, producing highly coloured substances, and in the second to the formation of insoluble oxidation products.

The effect of numerous solvents was examined. The most active was carbon tetrachloride, followed by chloroform and amyl alcohol. Straight-run spirit, ether and acetone were almost inactive in producing these changes. A mixture of 5% of carbon tetrachloride with ether is, however, as active as pure carbon tetrachloride. The alteration is very much more rapid in the mercury-vapour light than in daylight. In ultra-violet light and in the absence of oxygen, the yellowness produced was found to be proportional to the time of exposure. Oils of different origins were compared, of those examined Texas being the most or Russian the least affected. E. B. E.

See also Abstracts Nos. 746, 751, 764.

Anti-Detonation.

815. Behaviour of Anti-knocks. A. Egerton and L. M. Pidgeon. *Nature* 1931, **127**, 591.

The authors have shown that a small quantity of lead tetraethyl vapour, when let into an evacuated vessel heated to 265° C. into which a charge of pentane and oxygen is afterwards introduced, will not appreciably affect the course of the combustion and may even accelerate it. If oxygen is let into the vessel before the lead tetraethyl and then the bulk of the charge added, the combustion is invariably inhibited. Thus it is shown that the lead must first be oxidised before it is effective as inhibitor. C. C.

815a.—Detonation Characteristics of Some Aliphatic Olefine Hydrocarbons. Wheeler G. Lovell, J. M. Campbell and T. A. Boyd. *Ind. Eng. Chem.*, 1931. **23**, 555-558.

The relative tendencies to knock in an engine have been measured for twenty-five olefine hydrocarbons. The engine used and method of operating it have been described in a previous paper by the authors (*Ind. Eng. Chem.*, 1928, **20**, 1045). The measurements were made on the hydrocarbon in admixture with gasoline, the concentration used being one gram-molecule per litre; the results were expressed in terms of aniline equivalent. It was found

that compared with the corresponding paraffins, the straight chain olefines showed less tendency to knock, this tendency increasing with increasing molecular weight. It was also observed that in a homologous series of olefines (1) the tendency to knock increased with increasing length of the saturated carbon chain; (2) that in an isomeric series the tendency to knock decreased progressively with increasing asymmetry in the molecule and (3) that the tendency to knock is roughly determined by the length of the largest saturated carbon chain. The differencies between the knocking tendencies of the branched chain olefines and the corresponding paraffins do not appear to be constant. There are indications that the introduction of a double band so as to break up the largest chain of CH_2-groups has the greatest effect. The effect of introducing a second double band was examined in four isomeric hexadienes (diolefines). The results obtained agreed well with those expected from the known behaviour of the olefines already considered. S. F. B.

Engines.

816. Oil-Cooling and Oil-Coolers [in Internal-Combustion Engines]. W. R. Ramsaur. J.S.A.E., 1931, **28**, 429–434.

The recent increases in horse-power, compression pressure and engine speed have imposed additional duties on the lubricating-oil, with increased risk of troubles resulting from high oil-temperatures, of which the following are noted: excessive oil-consumption, smoking at the exhaust, fouled spark plugs, excessive carbon formation, engine wear and bearing failure. Bearing failure is considered the most serious. Babbitt metal rapidly loses its strength with increased temperature, and the bearings may crack and pound out long before the melting-point of the alloy is reached. Temperatures in excess of 300° F. have been reported in road operation; these should be reduced to about 210–230° F. for safety.

The function of an oil-cooler is to warm up the oil when cold (to facilitate lubrication while starting) and to cool it when hot. The cooler should not unduly restrict the flow of oil, should withstand continuous pressure and be easy to clean. The air-cooled type is used mainly on air-planes and the water-cooled type on automobiles. They have been shown to be reliable and satisfactory in operation.

Descriptions of coolers and results of tests are given. It is recommended that the cooler be cleaned out with benzol after every 20,000 miles operation. E. B. E.

817. Deeble Duplex Engine. *Mech. Eng.*, 1931, **53**, 294; N. French, Aero Digest, 1931, **18**, 76.

The chief feature is the double-acting principle, accomplished by employing a piston within a piston, the inner one being stationary. The first experimental engine was a twin-cylinder motor-cycle engine remodelled. Tests are to be conducted shortly with a six cylinder inverted in-line air-cooled engine, calculated to give 300 h.p. at 2000 r.p.m. W. A. W.

818. Frichs Diesel Engine. *Mech. Eng.*, 1931, **53**, 293–294; Brit. Motorship, 1931, **11**, 501.

This engine is of trunk-piston design, airless injection, rated at 800 i.h.p., although normal output is 700 i.h.p. at 235 r.p.m. At 720 i.h.p. at 235 r.p.m. mean indicated pressure was 7·2 Kg. per sq. cm., mechanical efficiency was 76·5% and fuel consumption equivalent to 0·41 lb. per b.h.p.hr. W. A. W.

819. Means Within the Cylinders of Internal-Combustion Engines for Vaporising and Mixing the Charge and for Permitting the Alternative Use of Light and Heavy Fuels. C. Bugaud. E.P. 340,005, 16.12.30. Appl., 16.8.29.

The invention provides a means for running low compression i.-c. engines with spark ignition on either light or heavy fuel using only one carburettor. This is effected by placing in the cylinder over the admission valve a hollow body with a series of helical shaped passages extending from its inner to its outer surface. The pitch of these passages is such that the jets of combustible mixture reach the cylinder walls at the circumferential circle corresponding to the bottom point of the piston's stroke. The hollow body mixes and heats the fuel, and thus the engine can be run without any pre-heating of the heavy fuels and without producing carbon deposits on the cylinder walls. The type of fuel used can be instantly changed whilst the engine is running at any load. J. M. F.

820. Means for Periodically Introducing Pulverulent Fuels into Internal-Combustion Engines. H. Holzwarth. E.P. 340,709, 8.1.31. Appl., 13.11.29.

The usual construction for admitting a measured quantity of pulverulent fuel to an engine relies on the use of piston valves moving from a measuring chamber to the atomising valve or combustion chamber. A film of fuel is deposited between the sliding parts which eventually causes seizure and breakage of some of these working parts. This invention employs a means using line contacts which removes this tendency. Means are also provided whereby the change of direction of the fuel stream is very small, thus eliminating any slight increase of pressure which tends to the caking of the fuel particles to form solid pieces. J. M. F.

821. Liquid Fuel Pre-heater and Vaporiser Attachment for Engine Carburettors. C. J. Law. E.P. 340,719, 8.1.31. Appl., 18.11.29.

The invention provides a system of heating fuels such as kerosine between the supply tank and the carburettor, and of heating the mixture between the latter and the inlet manifold of an internal-combustion engine.

Both heaters make use of the heat in the exhaust gases, and further heat is supplied to the mixture heater by an electrical resistance. A two-way valve is provided, so that the engine can be started on petrol to warm up the engine and the fuel heaters. J. M. F.

822. Method of Charging Four-stroke Airless-injection Diesel Motors. Motorenfabrik Deutz A.-G. E.P. 341,703, 22.1.31. Appl., 10.1.30.

Owing to the low pressure necessary in the blower for charging airless-injection 4-stroke Diesel engines, some of the exhaust gases pass over into the air blast whilst the inlet and exhaust valves are simultaneously open. Thus the purity of the scavenging and charging air is destroyed.

This invention provides means for avoiding this contamination by delaying the simultaneous opening period of the valves, so that it occurs substantially in the suction stroke. Then the pressure of exhaust gases in the cylinder at the opening of the inlet valve is lower than the blower pressure, so that the back flow of the exhaust gases does not take place.

Although the scavenging period is shortened, sufficient cooling and scavenging takes place as there is no to-and-fro flow of the exhaust gas in the cylinder. J. M. F.

823. Mechanical Injection Diesel Engines. V. E. Feeny. E.P. 341,945, 22.1.31. Appl., 22.10.29.

The invention provides means of improving and simplifying the construction and operation of regulating devices for Diesel engines. The length of spraying period, the quantity of fuel injected and the pressure maintained are regulated by a governor. Manual controls independent of the automatic controls and of each other are fitted to the injection valve and the fuel pump to facilitate starting. The governor is provided with a manual control, so that it can be made to regulate the injection and quantity of fuel delivered to the spray nozzle over a wide range of engine speeds.

This mechanism provides synchronous co-operation of the variable discharge pump and the spray nozzle, and variations in the fuel pressure to suit the output. J. M. F.

824. Method and Apparatus for Carburetting in Connection with Internal-Combustion Engines. Moore Inventions Corpn. E.P. 343,063, 9.2.31. Appl., 7.8.29.

The invention relates to an improved form of carburetting by better controlling of the flow of fuel introduced into the air stream at the engine side of an air control throttle. The flow is controlled in direct relation to the static pressure of the exhaust gases. The fuel is further vaporised by discharging combustion gases, the amount varying with the charge passing through the cylinders, at high velocity into contact with the fuel at its point of discharge. The mixture is further vaporised by impinging on a surface heated by exhaust gases. For high intake depressions supplementary air is bled with the fuel, and the fuel passage area is mechanically controlled for starting and idling. With this arrangement engines can be run on fuels of high b.pt. as well as the usual fuels, and all the fuel is put through a simple nozzle. Compensating jets and mechanisms employed in the usual types of carburettors can be eliminated, whilst the over-all efficiency of internal-combustion engines can be generally increased. J. M. F.

825. Two-Stroke Internal-Combustion Engines. W. Knockhenhauer. E.P. 343,131, 11.2.31. Appl., 11.11.29.

The invention relates to that type of engine with a piston compressor for producing compressed and highly heated combustion air which is forced into the working cylinder during the commencement of the fuel and compressed air charging strokes. The compressor cylinder is coaxial with the working cylinder, they being placed end to end and their pistons connected to a common crankshaft. The air is compressed in two stages, the first in the working cylinder, on the side of the piston remote from the combustion space; the second in the compressor cylinder, which has a very short stroke. The fuel is heated up to its ignition temperature before injection into the hot compressed air jet, so that there is no retardation of ignition and combustion is complete. J. M. F.

See also Abstracts Nos. 752, 753.

Coal and Shale.

826. Low Temperature Coal Distillation Plant at Dunston. *Engineering*, 1931, **131**, 368–389, 459.

A unit is described capable of distilling 60 tons of coal per day, and the important feature of the plant is that the hot semicoke is quenched before delivery to the stokers or pulverisers, thus enabling 50% of the steam required to be obtained very cheaply.

The actual process is the exposure of a shallow bed of fuel to a mixture of hot gases and steam, and the general arrangement is as follows. After screening, the coal greater than ¼ in. mesh is delivered to a conveyor and is discharged thence through a rotary valve into the retort, where a scraper conveyor distributes the feed evenly. Six rollers, on which are mounted helically arranged star-shaped rotors, serve to discharge the resultant semi-coke on to the quencher conveyor belt. These extractor rollers are actuated by a hydraulic system, which is automatically controlled in speed by the level of the coal in the retort.

The semicoke is quenched and then conveyed to the bunkers, meanwhile being exposed to a current of warm air which serves to carry off water vapour and other gases released by the coke in cooling.

The whole of the gas and vapours pass through a series of filters to remove entrained dust, and are then condensed in a water-tube condenser, the crude tar being treated in a steam heated Muddiman still to remove water and the lighter oils. The hot residue is pumped into a second still, producing creosote and pitch. After settling the creosote is redistilled with steam at a temperature of 200° C. Refined oil is so produced, yielding a higher priced product, and the residue is run off as heavy creosote.

The crude spirit is rerun to produce an unrefined motor spirit, which is treated with soda and acid, and thence it passes to a finishing still. White spirit, motor spirit, sodium cresylate and cresylic acid (crude) are obtained. Figures are given of the yields.

The cost of operating a 60 ton unit is estimated to be 16·9d. per ton of coal carbonised without by product recovery, and 30·8d. per ton of coal carbonised when refining processes outlined above are added. H. C. R.

827. Destructive Hydrogenation of Coal, Tars and Oils. I. G. Farbenind. A.-G. E.P. 344,165, 5.3.31. Appl., 31.1.30.

For the destructive hydrogenation of carbonaceous materials, large quantities of hydrogen are necessary, which hitherto have been obtained at considerable cost.

It has already been proposed to subject the raw materials to destructive hydrogenation and cracking in any order of sequence and to recycle the hydrogenating gas.

It has been found that the hydrogenation may be carried out simply and advantageously by subjecting the raw material to a cracking and a hydrogenation in any order of sequence, and employing the hydrogen which is obtained by treatment of the waste gases from the process by treatment in the electric arc. By this treatment of the waste gases the whole or the greater part of the hydrogen necessary is derived from the raw materials. Also, in addition to hydrogen, products such as acetylene and carbon black ethylene is formed.

After removal of the carbon black, ethylene, acetylene, etc., may be separated by washing under pressure with suitable solvents such as cyclo-hexanone, leaving the gas rich in hydrogen for employment in the hydrogenation process. R. P.

828. Low Temperature Distillation or Carbonisation of Carbonaceous Materials. C. Honnay. E.P. 344,301, 5.3.31. Appl., 23.5.30.

A muffle is used in which scrapers are fitted obliquely to an endless conveyor enabling a layer of material from 15 to 20 mm. in thickness to be propelled and ploughed on a heated hearth. The vapours distilling off pass through a filter designed to trap dust and vacuum can be used to assist in the distillation. It is claimed that the tar obtained contains less than 25% tar-acids, and that the percentage of coal dust in the tar does not exceed 15%. H. C. R.

829. Hydrogenating Carbonaceous Materials. I.C.I., Ltd., R. Holroyd and C. Cockram. E.P. 344,975, 19.3.31. Appl., 28.12.29 and 23.10.30.

The invention relates to destructive hydrogenation of carbonaceous material, such as bituminous coal, in the liquid phase.

A mixed catalyst is employed, consisting of a plurality of metals separately present in massive form, of which one is a member of the copper group or a member of group 2, while another is a member of group 3, 4, 5, 6 or 7 of the periodic system.

By catalyst in massive form is meant catalysts in a compact form, exposing a relatively small surface, such as balls, wire, ribbon, gauze or grids of metal. Thus silver or copper in the massive form, together with tin in the form of tin plated iron. Silver is preferable to copper as the former is more resistant to sulphurous impurities. These catalysts have the property of deoxygenating the carbonaceous material, thus inhibiting the formation of phenolic compounds. The ratio of the surface area of the tin plate to the copper or silver sheet is of the order 3 : 1.

Using a suspension of 30% Bentley coal in 70% of heavy oil, in the presence of hydrogen at 250 atm. and 390° C., and either copper or silver in conjunction with tin plates, yield of oil is about 74%, calculated on the coal.

R. P.

830. Destructive Hydrogenation. H. W. Strong and I.C.I., Ltd. E.P. 345,305, 20.3.31. Appl., 20.12.29.

This process of hydrogenation of materials, such as coal, oils, tar or the like, is carried out in the presence of nickel or cobalt alloyed or coated with tin. The nickel or cobalt may be alloyed with iron or with each other, but are further alloyed or coated with tin according to the invention.

Suitable alloys comprise the following: cobalt tin, nickel-tin, iron-nickel-tin, iron-cobalt-tin, etc., and these alloys may be used in the powdered or in the massive state. The alloy, however, should not melt below the ordinary conditions of working, which, in the liquid phase, may be 300°–450° C. at 20 to 500 atm., and in the vapour phases 400°–500° at pressures from atm. to 200 atm.

R. P.

831. Distillation of Coal Tar. Barrett Co. (U.S.A.). E.P. 345,654, 23.3.31, Appl., 21.11.29. Conv. (U.S.A.), 30.11.28.

A method of separating the volatile constituents from coal tar is described which consists essentially of a continuous distillation combined with blowing with an inert gas such as air. Means are indicated whereby the inert gas is circulated in a closed cycle and the heat abstracted by various alternative methods.

H. C. R.

832. Separation of Oils from Solid Substances. M. Pier, R. Wietzel, B. Engel, Assignors to I. G. Farbenind. A.-G. U.S.P. 1,794,865, 3.3.31. Appl., 17.8.28. Conv. (Ger.), 25.10.27.

Mixtures of charred residues, ash or catalysts are obtained in the heat treatment of carbonaceous materials, and it is often difficult to separate the oil from the solids.

If aromatic hydrocarbons such as benzene, toluene, xylene or other suitable solvent be mixed with the oily residue, and the mixture centrifuged, separation is easily effected. The operation may be carried out in stages—*e.g.*, a quantity of sludge from the destructive hydrogenation of brown coal is centrifuged at 130° C., and an oil free from coal is obtained, but the material still contains 40% of oil. To this residue is added one-third to one-half its weight of heavy benzine, resulting from the hydrogenation process, and centrifuging is continued leaving only 3–5% of oil in the remaining solids.

R. P.

ABSTRACTS.

Geology.

833. Petroleum in Uganda. Summary of Progress of the Geological Survey of Uganda, 1919-1929. E. S. Wayland. 1931. Pp. 26-27.

Later investigations in the area north of Kibiro have added five seepages to the three (Kibiro, "Mawa" and Kibuku) known in 1925, one 5 and one 14 miles south of "Mawa," two in the Ngeti country, and a small intermittent seepage at Kibiro on the Bunyoro shore.

An analysis of the Kibiro asphaltic seepage is 56·8% soluble in acetone, 42·5% soluble in chloroform, 0·07% ash, and 0·31% sulphur. The calorific value is 18,598 B.Th.U.

The oil, when dried, had a sp. grv of 0·961 and gave 1·1% to 150° C., 12·4% at 150°—300° C., 55·6% above 300° C., and 30·9% coke and pitch.

A sample of the Kibuku seep was examined and the oil extracted had a sp. gr. of 0·906, a f.pt. of 114° C., and was characteristic of a crude oil which had lost its lighter constituents by exposure.

Studies of the petroliferous area of the Albertine depression have recommended exploration by drilling. G. S.

834. Search for Petroleum in Italy. *Rev. Petrolifere*, 1931 (424), 621–623.

Petroleum prospecting was begun in Italy in the belief that under the alluvial cover of the Adriatic slopes of the Appennines, and the Po Valley, there existed a series of gentle, regular anticlines. It was proved subsequently, however, that the structures were far more complex than had been anticipated. Prof. Anelli considers that an undulatory seafloor was present in Pliocene times, which corresponds to fault zones (functioning probably during the Quaternary) giving rise to diapir structures, and permitting the ascension and accumulation of oil in suitable areas. This theory has since been well borne out by drilling and geophysical work, notably on the Montepelato-Marano anticline of 1928. The latter has been mainly carried out by magnetic and gravimetric methods, sometimes employed simultaneously, the torsion balance revealing the Fontevio and Carriago anticlines, and the magnetometer the Montepelato. The variable and feeble physical constants, and the complex structures, make the greatest precision essential to success.

The future of the fields would appear to fully justify the tenacity of the searchers. A. L. G.

835. Petroleum in Italy. L. Dompe. *L'Ingegnere*, 1931, **5**, 174–175.

For some time past petroleum has been known to exist in Italy, particularly in the Emilian region (provinces of Parma and Piacenza). Specially deserving of examination is said to be the marginal area surrounding the Salsomaggiore Basin, which has recently been studied during trial borings made for investigating the iodine-salt content of waters.

The formations include Eocene, Miocene, Pliocene and Quaternary. The Eocene consists essentially of clays and marly limestones with inclusions of sand. The Miocene occupies the greater portion of the basin, and is composed of thick alternations of clayey and sandy marls, limestones and sandstones. The Pliocene borders the basin on the south, and is made up of sub-appenine material consisting of conglomerates with gneiss and granite fragments, blue marls and sand, while the Quaternary consists of the alluvium of Ghiara.

Reference is made to borings, the chief of which is the Salsomaggiore boring which has yielded increasing quantities of salt, with a notable production of oil. Further accurate observations on this boring, however, are considered desirable, and the deepening and widening of it are suggested.

All analyses of waters from the Salsomaggiore area show that the composition is very different from that of sea water, and it is evident that the presence of oil and gas is not accidental but is related to this peculiar composition.

G. S. S.

836. Petroleum Deposits of Japan. *Petr. Times*, 1931, **25**, 720.

The oil is found mostly in anticlinal structures parallel to the coast. Liparite, basalt and their corresponding tuffs are found in the deepest measures.

Kiu-hei Kobayashi considers that the petroleum is derived from fish oils: volcanic action beneath the sea kills and buries the fish. The proteids and bones are destroyed, and the oils, being more chemically stable, are set free and float to the surface of the water. These are then carried by wave action to a neighbouring shore where they are absorbed by volcanic muds consisting mainly of acid clay. Elutriation by wave motion causes the light particles of the clay to come to the surface, the coarse material sinking to the sea bed. If this bed is later subjected to geothermal action, the fish oils may be transformed into petroleum vapour, which escapes through fissures to overlying porous sedimentary deposits. This view explains the absence of fossil fish in the present oil measures.

M. R. B.

837. On and Off the Dome at Kettleman Hills. S. C. Herold. *Oil Weekly*, 1931, **61** (3), 36.

In considering the life of this field the author points out that at present this is bound up with the question of the thickness of the Temblor formation, the economic limit of drilling depths, and the possibilities of faulting, subsidiary doming and overlap on the flanks of the proved structure. There is considerable evidence that one of these features is present in the area, and in this case a considerable chance of an enhanced zone of production. The author concludes that any attempts to determine the trend of production at present are mere guesswork in the absence of adequate data.

V. C. I.

838. East Texas Oil Yield Expected to Exceed Billion Barrels. J. Logan. *Oil Weekly*, 1931, **61** (4), 25.

Expectations of a high yield of petroleum in East Texas are based on the possibility of an extension and thickening westwards of the oil sand. The producing sand, the "Woodbine Sand," pinches out to the east on an old Cretaceous shore line along the Sabine uplift, thereby trapping the oil. The sand dips gently and regularly westwards to the bottom of the east Texas basin. Production will therefore depend on the edge-water limit. It is expected that Joiner, Kilgore and Longview pools will merge into one continuous field.

P. A. S.

839. History-Geology-Mechanics, Ritz-Garrett-Decker Area. F. Swindell, *Oil Weekly*, 1931, **61** (5), 30.

In this field the upper producing horizon is the Mississippi Lime, and is an old eroded surface of pre-Pennsylvanian times encountered at about 3000 ft. Production in the lower horizon is from a break in the Viola lime, which is sandy, at about 3400 ft. The structure has not been fully exploited, but it is thought that the crest runs N.E.—S.W., placing the Ritz Pool and the Garrett-Kaegi production on one high and the Decker gas field on a separate structure, running parallel to it. P. A. S.

840. Bacterial Genesis of Hydrocarbons from Fatty Acids. L. A. Thayer. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 441–453.

On the assumption that petroleum has been formed in Nature from organic remains the author discusses the decomposition of the fats, fatty acids, and alcohols by bacteria. He points out that the evidence which has been collected indicates that all such compounds are reduced to carbon dioxide and methane by bacterial decay. Further research work has been done on salts of various organic fatty acids intercalated with muds from different environments. The results confirm the conclusion that the anærobic decay of saturated fatty acids by bacteria leads to the production of methane as the only hydrocarbon, and no evidence is forthcoming of the production of higher hydrocarbons by this method of attack. V. C. I.

841. Effects of Underground Storage Conditions on Characteristics of Petroleum. P. W. Prutzman. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 455-464.

The author bases his theory on the supposition that petroleum is produced by the destructive distillation at a relatively low temperature of organic matter disseminated in sediments. The mixtures of hydrocarbons thus formed would be subjected to distillation and recondensation in the crust and would therefore be modified by the following processes: dissociation and reconstitution of the crude as a result of heat and pressure; fractional distillation as a result of unequal heat distribution; selective filtration in passing through the strata of the earth's crust; modification by the attack of sulphur disseminated throughout the crust in small quantities. The author concludes that it is the combination of these processes rather than the differences in original material which have produced the diversities in the known types of petroleum. V. C. I.

842. Development and Production History on the Salt Flat and Other Fault Oilfields of East Central Texas. H. B. Hill, E. V. H. Bauserman and C. B. Carpenter. U.S. Bur. Mines Rept. Invest. 3050, 1931.

The Salt Flat Field, Caldwell Co., Tex., is about 5 miles east of the old Luling Field, and has a proved area of about 1280 acres. To Sept., 1930, 332 producing wells had been drilled, the majority obtaining oil from the Edwards limestone at about 2700 ft. The field is a fault structure, the strike being approximately N. 20 to 45° E., the displacement being probably 200 to 500 ft.

Data on water production show that the average per well increased from 30% for Jan., 1929, to 81% for Dec., 1929. In the old Luling Field the daily water production in July, 1929, was about 150,000 barrels, as compared with 11,000 barrels of oil. G. S.

Field Technology.

843. WELL PRESSURE FURNISHES POWER FOR SNUBBING TUBING. J. Logan, *Oil Weekly*, 1931, **61** (3), 26.

The running of tubing in wells which are not producing satisfactorily has been the means of improving conditions in the Barber Hill Field, Texas.

The tubing was run in without killing the well and at no time was production interrupted. The pressure from the well was used to snub the tubing in; thus the equipment was operated automatically.

The tubing was plugged at the bottom with two plate glass discs which were broken by dropping a godevil after the tubing had been run.

The tubing control head was secured to a swedge in the 8¼ in. master gate valve, and above this head was placed a snubbing head. Above the snubbing head was placed a snubbing cylinder, and a pressure intensifier was located on one side and connected by piping to the tubing control head behind the rams. The snubbing head and snubbing cylinder were operated directly by the well pressure, and the control head was operated by an intensifier which in turn was operated directly from the well. These 3 units, operating directly from the well, were controlled by 3 4-way valves operated by one man in a stationary position. The intake side of the manifold was connected to the well below the tubing control head and the exhaust side of the manifold was connected into the flow line to the separator.

The tubing control head was in the form of a cross, and the rams could be operated manually or hydraulically.

The snubbing head was also in the form of a cross, and the body had an enlarged bore at each end of the horizontal axis. A piston was placed in each of these enlarged bores and was secured directly to the rams. The well pressure was piped to each side of the pistons and could be diverted to either side by the four-way valve. Opening and closing of the rams was performed at will by the diversion of the pressure to the front or rear of the piston. At the top of the snubbing head was a bowl equipped with slips which were held up by a strong spring. These slips held the tubing in the well as it was snubbed in while the travelling slips were run up to get a fresh grip on the tubing.

The snubbing cylinder was mounted above the snubbing head on a column of steel rods. The hollow piston had a stroke of 4 ft. and would snub in a maximum of 4 ft. of tubing at each stroke. The hollow piston rod was 5½ in. outside diam. and the tubing was fed through this rod. To the lower end of the hollow piston rod were attached an inverted bowl and a set of snubbing-in slips which travelled up and down and gripped only on the downward journey.

The pressure intensifier consisted of two opposed unequal cylinders with interconnected pistons, the larger one connected directly to the well below the tubing control head and the smaller one to the control head behind the rams. Pressure entering the larger cylinder actuated the piston in it, which in turn actuated the piston in the smaller cylinder. The smaller cylinder was filled with liquid which was forced through the piping into the control head behind the rams. Due to the varying diameters of the cylinders this liquid was forced, at a higher pressure than that in the well, and it closed the rams against the well pressure. The well pressure opened the rams in the tubing control head when pressure on the larger piston in the intensifier was released. As the rams opened, the liquid behind them flowed back into the intensifier.

With the snubbing head and tubing control head open and the master gate closed the tubing was lowered through the tubular piston rod. With the lower end of the tubing just above the gate valve the snubbing head was closed around it and the gate valve was opened and the process was begun. The piston in the snubbing cylinder was run to the top and then brought down, snubbing in four feet of tubing. This operation was continued until the coupling was just above the rams in the snubbing head. The rams in the tubing control were then closed and those in the snubbing head were opened. As soon as the coupling had passed the snubbing head rams, these rams were closed and those in the tubing control head were opened. This operation was continued until the weight of the tubing obviated the necessity for snubbing. At this point the snubbing cylinder was removed and the tubing was lowered with elevators. L. V. W. C.

844. Bottom Hole Pressure in Oil Wells. C. V. Millikan and C. V. Sidwell. *Int. Petr. Tech.*, 1931, **8**, 213.

The determination of the most efficient methods of recovery and lifting procedure can only be made when a definite knowledge of the pressure at the bottom of an oil well, at any existing operating conditions, is known. In addition the relation of this pressure to the pressure in the producing formation should also be known.

Several methods exist for calculation of bottom hole pressure. The fluid head will give it where the well is inactive, or the addition of the casing head pressure, the static head of gas, and the fluid head will determine it when the well is shut in.

For wells flowing naturally through tubing the pressure at the bottom of the tubing may be calculated by adding the pressure at the casing head between the tubing and the casing and the pressure due to the weight of the column of gas, but there is always a possibility of error caused by fluid being in the annular space above the bottom of the tubing.

Probably the most accurate method of calculating bottom hole pressures is by adding to the pressure at the tubing head the pressure due to the weight of the column of gas.

Several types of pressure bombs have been used with varying success, and recording gauges have also met with a certain amount of success. In the determination of the bottom hole pressures considered in this paper the Amerada Pressure Gauge was used and consisted of a clock, chart carrier and pressure element. The movement of the chart is obtained by a central screw operated by the clock, and this screw drives the chart carrier downward so that its weight almost balances the friction thus reducing the power demand on the clock. The pressure element consists of a pressure element tubing in the form of a spiral coil, and the lower end of this tubing is joined to an opening in the base which extends to the outside of the bomb. The upper end is sealed and attached to a shaft to which is attached a recording stylus. Five different speed clocks are obtainable, and pressure elements ranging from 75 to 1100 lbs./in. of movement of the stylus may be used. A number of charts are given showing the results of tests under varying conditions.

An examination of the pressures met with at different depths permit of certain precautions being taken which might otherwise be overlooked. Numerous tests were made and it was shown that the correlation between casing head pressure and bottom hole pressures was often indefinite and becomes more irregular as the bottom hole pressure approaches the pressure in the producing formation. As a result of these investigations the authors have concluded that by comparing bottom hole pressures and pressures

within the producing formation it is possible to determine whether the rate of production being obtained is limited by the capacity of the method of lifting the oil or by the capacity of the well to produce.

The best size of flow string for a well flowing naturally or by gas lift must be determined by the calculated bottom hole pressure, and the degree of accuracy is in proportion to the accuracy of the bottom hole pressure upon which the calculation is based. Regulating the pressure at the casing head or changing the operating method will usually ensure a more efficient use of the gas energy accompanying the oil, retard bottom water invasion or more effective natural water flood. L. V. W. C.

845. Flow of Saturated Oil at Back Pressures above Saturated Pressures. W. F. Cloud and I. F. Bingham. *Oil Weekly*, 1931, **61** (10), 24.

An effort was made to determine whether or not saturated crude oil can be made to flow as an unsaturated or dead oil, and, if so, whether the rate and amount of recovery will be practically the same as that of unsaturated oil.

It has been supposed that a saturated oil flows more readily through a porous formation than does an unsaturated oil, and this supposition was based upon the theory of reduced viscosity as affecting flow, due to the gas in solution, without considering such facts as plugging of the pores of the formation by bubble growth.

Back pressures were used above the pressure at which the oil was saturated, thus preventing the gas from coming out of solution, and so eliminating retardation of flow due to the presence of gas bubbles between the sand grains. Curves were plotted showing recovery against various pressure differentials, and it was found that at the highest differential used the curve was a straight line that, if extended, would pass through the point of origin.

In addition, in the region of two lower differentials the two curves cross the straight line curve, and if these are extended they would not pass through the point of origin. This may be due to the greater viscosity of the dead oil which produced the curves of the lower differential. The three curves are closely similar, and it would seem that a condition of flow has been established where a live or saturated oil flows as a dead oil. This is probably due to the fact that gas is prevented from coming out of solution, and thus prevents the Jamin effect in the sand. The authors arrived at the following conditions :—1. Other conditions remaining constant, dead oil flows faster through a porous formation than does a saturated oil 2. Unsaturated oil flows without bubble growth. 3. With sufficient back pressure saturated oil flows more efficiently, and with lower gas-oil ratio than it would do with wide open flow. 4. The experiment appears to prove that saturated oil may be compelled to flow as dead oil by maintaining discharge pressures on flowing wells equivalent to original saturation pressures, building up, of course, differential pressures above the initial formation pressure by injection of gas through key wells. 5. Provided the injected gas does not go into solution readily the differential, theoretically, has no limit, provided discharge pressures are maintained equal to, or greater than, the pressure at which the oil was saturated. 6. It has been well established that gas is not readily soluble in crude oil without considerable agitation. L. V. W. C.

846. Bottom Hole Choke Decreases Gas Oil Ratio. H. C. Otis. *Oil and Gas J.*, 1931, **30** (1), 64.

The purpose of the tests being made with bottom hole chokes is to determine the increased efficiencies to be obtained by adjusting flow velocities within the tubing. A reduction in gas-oil ratio is thought probable.

The combination of large diameter tubing and restricted production under proration has resulted in high delivery pressures and low flow velocities, with a consequent high slippage factor. The use of stop-cocks and smaller tubing has resulted in some improvement, but such methods have not met with general approval.

Bottom hole chokes have an advantage over surface chokes, since by the first means the gas is forced to expand in the lower portion of the tubing, and the energy normally lost between the surface choke and the trap is used in lifting the oil. The small volume occupied by each cubic foot of gas resulting from the high pressure at the inlet of the surface choke causes a low flow velocity. By choking on the lower part of the string and delivering the wells allowable production at the surface at or slightly above atmospheric pressure permits the gas to occupy a larger space than it would otherwise do under a high delivery pressure, and an increased flow velocity results. Conversely, it is possible by bottom hole choking alone to raise inefficient low velocities to equally inefficient high velocities. A flow velocity which is too high will result in a loss of maximum efficiency, but through the combined use of a bottom hole choke and a low pressure surface choke, tubing flow velocities may, within certain limits, be regulated almost as desired for any rate of production. A variation of the delivery pressure over a range of only a few pounds with the surface choke, provided the delivery pressure is close to atmospheric pressure, enables the regulation of tubing flow velocities to be accomplished. The varying of a few pounds in delivery pressure, providing the delivery pressure is near atmospheric, will have little effect upon the velocity through the sand, but will have a marked effect through the tubing.

By securing a more efficient velocity will have an indirect effect upon the velocity through the sand due to a lowering of the gas-oil ratio. A combination of bottom hole and surface choking at a few pounds above atmospheric provides a very sensitive control of tubing flow velocities independent of sand velocities.

The choke used in the tests on the Texas fields is run in on a Halliburton steel line, and can be set at any desired depth. Beans of various sizes can be used, and the operation of changing from one size to another is a simple and quick process.

These experiments have not been conducted for a sufficient length of time to give any very definite data, but the results to hand indicate that bottom hole choking will undoubtedly be included in future operating programmes.

L. V. W. C.

847. Bending Force Affects Threaded Joints. J. T. Hayward. *Oil and Gas J.*, 1931, **30** (2), 14

The object of this paper is to explain the tendency of rotary tool joints to unscrew when the direction of rotation should apparently cause them to make-up.

If a short length of pipe is gripped in the jaws of a chuck with its axis horizontal, and another length of pipe of slightly larger diameter is allowed to slide off the first pipe, this second pipe will incline slightly downwards, the two pipes making an obtuse angle of a little less than 180°.

If the outsides of the two pipes are marked in some way and the chuck slowly started, it will be found that the pipe held in the chuck will rotate at a slightly faster speed than the pipe not held. The two pipes are in rolling contact at the points where the end of the held pipe touches the inside of the loose pipe at its upper edge and where the end of the loose pipe touches the held pipe at its lower edge, the pitch circles being the external diameter of the

inner pipe and the internal diameter of the outer pipe. As the two differ the angular velocities will differ, and there will be a relative angular movement between the two members.

If now a third piece of pipe of smaller diameter is slippedi nside the larger pipe and the clamped pipe again rotated, it will be found that this third pipe will rotate slightly faster than the second pipe. This arrangement corresponds to the regular collared pipe joint. If the two parts are tapered a similar action will result, provided the tapers correspond.

A similar tendency will maintain if the pipes are threaded and screwed together, provided the deflecting force is sufficient to produce play in the joints. If the threads are right hand and the direction of rotation clockwise the top joint of the collar tends to screw up whilst the lower joint will tend to unscrew.

These conditions are found at rotary drilling wells when the holes are not straight. At any crooked part of the hole there will be a tendency for joints, with pin pointing down, to make up, and for those with pin pointing up, to unscrew. In a made up joint the difference in the diameters of the pitch circles is of course small, and the relative rotation is consequently also small compared with the total number of turns made by either piece.

In the case of the pin pointing up there is a possibility of the joint working backwards and forwards, since the tendency to unscrew is opposed by the torque transmitted by the column of drill pipe, and is also affected by the curvature of that part of the hole in which the joint happens to be. The movements will, of course, lead to undue wear, and will result eventually in a twist off. In addition, it must be remembered that the length and stiffness of the drill pipe are very large compared with the threaded parts, permitting a large leverage, and the tendency of the one part to rotate relatively to the other will consequently be large and will be able to overcome quite a big torque.

The Author concludes with the following suggestions:—1. Screwed joints with the pin pointing up should be avoided as far as possible in the drilling string. 2. Joints with the pin pointing up should be made up firmly before being used in the string. 3. The taper of the two engaging parts of a joint should correspond within very close limits. L. V. W. C.

848. Use of Aluminium for Oil Lease Tanks. I. Field Tests. L. Schmidt, J. M. Devine, and C. J. Wilhelm. *U.S. Bur. Mines Rept. Invest.* 3066, 1931.

Tests show that aluminium, on account of its resistance to corrosion by hydrogen sulphide, has possibilities for use in lease tankage. There are three definite limitations, as follows:—1. For steam coils where salt water is encountered, aluminium is not practicable, but it is possible that other suitable forms or alloys may be developed. 2. Aluminium tank plate is not highly resistant to the action of concentrated brines. 3. When aluminium and steel are used in conjunction, no electrolyte must be present, and efficient insulation between the metals is necessary. Further tests are being made. G. S.

Crude Oil.

849. Heavy Oil in Creek County, Okla. Presents Interesting Problems. L. G. E. Bignell. *Oil & Gas J.*, 1931, **29** (46), 83.

The crude oil production from the Dutcher sand formation near Bristow, in North-Eastern Oklahoma, varies considerably in quantity and quality. Usually, the oil is light, gravity 29° to 34° Bé, but recently heavy oil with a gravity of 18·3° Bé was struck at 3300 ft. The analysis of this oil is: Asphalt

90%, Gasoline 6%, Kerosine 3%, B.S. & W. 0·02%, Sulphur 0·08%. It should be quite suitable without further treatment as road oil, owing to its high asphalt content.

The variation of the gravity of the oil in this field from 18 to 41° Bé has given rise to much speculation. It has been suggested that the original oil is light and has become oxidised while the lighter components have evaporated, localisation of the heavy oil being caused by the action of underground waters or by a break in deposition allowing erosion of the Dutcher sand which may have extended further west than well data now indicate. C. L. G.

850. Gasolines from West Texas Crude Oils. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (46), 30.

Crude oils from the Permian Basin in Texas, except those from the Yates and Hendricks Pools, which are naphthenic, have an intermediate or mixed base. West Texas crudes contain 1·25/2% of sulphur, which occurs as H_2S in large amounts, as elementary sulphur or as organic sulphur compounds. The untreated gasolines often contain 0·16/0·23% of sulphur, and require careful treatment to bring them to specification without destroying their anti-knock quality. The properties of the crude oils from the main fields are presented together with fractional distillation data, where available.

The yield and properties of the products obtainable from these crudes are tabulated and information on the distribution of sulphur and refining methods given in certain instances. Samples of mixed West Texas crudes, principally from the Yates and Winkler pools, were topped and the residues subjected to cracking. The properties of the topped crude oils, conditions of cracking, and the yields obtained are tabulated. About 40% of gasoline is obtained by the "flashed residue" method and 61% by cracking to coke.

The pressure distillate was refined with sodium plumbite acid and water, and then distilled with steam to produce finished gasoline and pressure distillate bottoms. The flashed residue possessed a low viscosity, pour point and sediment content, and a high calorific value. Analyses of the gases and coke are given. Kerosine distillate from the mixed crudes was cracked at pressures of 350/400 lbs., and temperatures of 948/950° F. yielding 70/72% of gasoline of 84/89 Octane No., 11·3/13·3% flashed residue and 688/752 cu. ft. of gas per brl.

The properties of the gasolines before and after treatment with, 4, 6 and 8 lbs. of acid per brl. are given. An alternative method of treatment is to distil off 30/50% of the gasoline, which requires no treatment, and then treat the bottoms with acid, water and soda and distil to E.P. The two fractions are then blended. Gasoline produced by the second method had the following properties: I.B.P. 93° F., 20% distilling over at 170° F., F.B.P. 358° F., Gum content 7 mg. per 100 c.c., Sulphur 0·07%, Octane number 86 in "Series 30" Ethyl engine. The properties of the flashed residues are also given.

R. A. E.

851. Kentucky Crude Gives Excellent Cracking Stock for Oil Refining. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (48), 140.

Kentucky crude oil is green in colour and has a high gasoline content. The average A.P.I. grav. is 32/38°; the heaviest crude (Grav. 22°) is found in Ragland County, and the lightest (Grav. 51·6°) in Johnson County. Crude oil from Western Kentucky has the following properties:—A.P.I. Grav. 35·0°, Sulphur content 0·33%, NaCl content 13·5 lb./1000 brl., straight run gasoline yield 33·6%, topped residue 65·0%. The straight run gasoline obtained had an A.P.I. Grav. of 59·5°, I.B.P. 110° F., 20% distilling over at

188° F., F.B.P. 410° F., Octane number 47. The topped residue (visc. Furol at 122° F. = 21 sec.) was subjected to cracking at 900° F. and 265 lbs. pressure, using the "flashed residue" method of operation. Three tests were made and the results obtained were :—Cracked gasoline yield 51·3/55·1%, flashed residue yield 47·0/44·0%, gas yield 231/336 cu. ft./brl. The properties of the cracked gasolines obtained were :—A.P.I. Grav. 58·0/61·4°, I.B.P. 82/92° F., 20% over at 178/192° F., F.B.P. 375/408° F., Octane number 77/75; The viscosities of the flashed residues ranged from 72/103 sec. Furol at 122° F. By blending the straight run and cracked gasoline in the proportions in which they were produced from the crude oil a total yield of 67/69% of gasoline having an Octane number of 65 was obtained. All Octane numbers were determined in a "Series 30" Ethyl knock-testing apparatus.

R. A. E.

852. Evaluation of a Crude Oil. W. L. Nelson. *Petr. Eng.*, 1931, 2 (7), 163.

To determine in the laboratory the yields and qualities of products that can be obtained from a crude on the plant scale, it is necessary to know (1) the true boiling point distillation curve (T.B.P.C.), (2) the flash vaporisation curve, and (3) the viscosity and gravity curves of the cuts obtained in the T.B.P. distillation.

A description of a laboratory still for determining the T.B.P. curve is given, together with curves of the viscosity, gravity and yields of fractions from a pressed distillate under examination. The apparatus used for determining the flash vaporisation curve is that described by Leslie and Good (*J. Ind. Eng. Chem.*, 1927). The conditions in this still correspond almost identically with plant conditions. By reading from the curves drawn up the yield and gravity of products of the required viscosity can be determined. This method is quite satisfactory for distillates, it being necessary to top crude oils or residues to remove the heavy tars. It is often very helpful to know the base of the oil being examined. This can be determined from the Bureau of Mines Key Fraction Numbers (see *Bur. Mines Repts. Invest.*, No. 2806). Thus, if the gravity of the fraction boiling between 482° F. and 527° F. is above 40° A.P.I., the oil is paraffin base, and if 33° A.P.I. or lower it is naphthene base. Similarly, a cloud point below 5° F. of the fraction boiling between 527° F. and 572° F. indicates a non-waxy oil of naphthenic type.

Other methods of obtaining an indication of the base of an oil—viz., by relation of yields of gasoline and gravity of crude, by viscosity temperature relationship are given with curves showing examples of different types of crude.

C. L. G.

853. Breaking Petroleum Emulsions. J. Hyman and A. F. Schlandt. Assrs. to Pure Oil Co. U.S.P. 1,800,837, 14.4.31. Appl., 26.3.29.

Acid anhydrides in the form of sulphur dioxide and hydrogen sulphide are introduced into the emulsion, and react directly to form lyophilic negative colloids. The emulsion is finally heated, and then allowed to stand until the separation of the oil and the aqueous constituents takes place. W. H. T.

854. Distillation. A. Schmalenbach, assr. to Koppers Co. U.S.P. 1,802,539, 28.4.31. Appl., 8.12.25.

Lower boiling-point constituents are separated from a mixture of liquids by first distilling the mixture to volatilise all the lighter bodies, which are condensed and collected in a vessel connected to a vacuum pump. The liquid siphons to a second distillation vessel at the bottom of a fractionating

column. The supply of heat to this distillation vessel and the abduction of heat from the analyser situated at the top of the column are so regulated that only solvent free from the desired dissolved substances leaves the bottom of the fractionating column. Vapours passing through the analyser are condensed and collected in a vessel which is also connected to the vacuum pump through a pressure-reducing device. W. L.

See also Abstracts Nos. 890, 915, 917, 922.

Gas.

855. Quick Method of Obtaining Gasoline Content of Casing-Head Gas. F. B. Taylor. *Petr. Eng.*, 1931, **2** (7), 85.

The apparatus described is a portable set for determining the approximate gasoline content of natural gas. It consists of a pressure regulator, orifice meter fitted with U-tube connections to enable the gas pressure to be read, a steel tube containing absorbent charcoal and venting to the atmosphere, and the necessary thermometers. Gas is passed through the regulator and orifice meter until temperature conditions are stable, the flow is then momentarily cut off by a stop-cock whilst connection is made to the charcoal tube. The gas is then turned on again for a definite period whilst the pressure differential in the U-tube, the gas temperature and atmospheric temperature are recorded. The altitude at which the test is carried out and the gravity of the gas are also determined. The volume of gas passed can be calculated from these data and the known orifice coefficient. The charcoal tube, which may be placed in an ice bath if necessary, is sealed immediately after the test and sent to the laboratory.

The charcoal is transferred to a 2-litre distillation flask, with the aid of glycerine. The distillation flask is connected to a water-cooled condenser, followed by a metal tank condenser filled with ice and delivering into a stoppered graduated receiver fitted with a vapour outlet. The uncondensed gases pass through this outlet to a graduated and cooled receiver containing heavy naphtha where the light gasoline is absorbed and the remaining gas vented. Distillation is carried on to 400° F., but the absorber flask is removed at 212° F. The outlet tube of the distillation flask and the succeeding condensers are set at an angle of 75° to the vertical, and bends in the condensers are avoided, to reduce drainage loss as far as possible. R. A. E.

856. Separation of Gases. Girdler Corporation. E.P. 344,786, 4.3.31. Appl., 4.12.29.

Acidic gases such as carbon dioxide, sulphur dioxide and hydrogen sulphide are separated from gaseous mixtures by means of an absorbent. The gases thus removed may be recovered, or the process may be operated for the purification of other gases. The absorbing agents used are substitution products of ammonia such as diethylamine or ethylamine or ammonia substitution products of alcohols, preferably triethanolamine. In the second stage the absorbing agent and the acidic gas are regenerated by decomposing the salt of the nitrogen base produced during absorption. A suitable form of apparatus is described.

The boiling point of the absorbent should preferably be higher than that of water. In the case of triethanolamine this compound may be used in a pure state or in solution. E. N. H.

Motor Fuels.

857. Ionisation in Gaseous Explosions. W. A. Kirkby. *J.C.S.*, 1931, 134, 878-885.

The rôle played by ionisation in gaseous explosions has been increasingly studied during recent years. Garner and Saunders have recently attempted to measure instantaneous values of ionisation during gas explosions, but their results were vitiated by the absence of the requisite conditions for critical damping of the galvanometer. This paper describes experiments similar to those of Garner and Saunders, but using a more accurately recording galvanometer circuit, which is described in detail. By means of this circuit, direct correlation was made of the progress of flame with the ensuing ionisation. Evidence has been obtained that "after-burning"—the re-illumination observed in the products of the initial combustion—is closely connected with recombination of ions formed during the primary reactions.

G. B. M.

858. Explosions in Closed Cylinders. Part IV. Correlation of Flame Movement and Pressure Development in Methane-Air Explosions. W. A. Kirkby and R. V. Wheeler. *J.C.S.*, 1931, 134, 847-854.

The combustion of the range of inflammable mixtures of methane and air has been studied by photographic means in closed cylinders of fixed diameters and varying lengths. In lengths exceeding 140 cm. it has been shown that the flame movement becomes vibratory even in mixtures initially at atmospheric temperature and pressure. The pressure fluctuations during the combustion have been correlated with the flame movement by means of an optical gauge of small inertia.

It has been found that the flame vibrations are due to a stationary longitudinal wave maintained in the column of gas behind the flame front. The maintenance of a "balanced frequency" of flame vibration is necessarily accompanied by a flame acceleration as the rate of heat release increases due to the higher initial compression of each layer to be ignited. The striations appearing in the partially burnt products behind the vibrating flame—striations which have been reported by several previous observers—are attributed to alternate release and recombination of ions as the pressure and temperature fluctuate in the vibrating medium.

G. B. M.

859. Factors in Treating Natural Gasolines. H. Martin. *Oil and Gas J.*, 1931, 29 (47), 57.

The two methods generally employed are (1) treatment with sodium plumbite and (2) treatment with sodium or calcium hypochlorite. Sodium plumbite is used for refining sour and high sulphur content gasolines, and sometimes gives a more stable product than hypochlorite, unless the latter is carefully controlled. It is also used for Government specification aviation gasoline when a neutral residue in the distillation flask is required.

It is estimated that 80% of the natural gasoline treated in the field is refined by the hypochlorite process which presents no difficulties with gasolines which possess a low sulphur content and require less than 2 lb. of chlorine per 1000 gal. treated. The gasoline may be bubbled through a tower containing hypochlorite, but when more than 1lb. of chlorine per 1000 gal. is required, contacting with the aid of orifice or jet mixers is recommended.

Whenever the consumption of chlorine is more than 25 lb. daily, or even less in cases where caustic soda must be employed because the gasoline contains hydrogen sulphide, it is preferable to use chlorine and caustic soda rather than bleaching powder. A new product known as H.T.H. (high test hypochlorite) is now available for use in place of bleaching powder. It possesses the following advantages: (1) 65% available chlorine instead of 37% max.; (2) greater stability on storage; (3) is not hydroscopic; (4) contains only 4% insoluble matter, thus giving less sludge and reducing cleaning operations; (5) 2½ times the volume of gasoline can be treated by the same weight of reagent; (6) more easily soluble. The manufacture of H.T.H. is described and information given regarding the most suitable conditions for preparing sodium hypochlorite solution. R. A. E.

860. Less Propane in Fuel indicated by Study of Vapour Lock Factors. F. C. Burk, *Oil and Gas J.*, 1931, **29** (43), 138.

Experiments have been carried out on a representative group of cars to ascertain the temperatures at which vapour-locking occurred, using calibrated thermocouples inserted at strategic points. The fuel systems investigated comprised gravity, fuel pump and vacuum types. Using a basic fuel which caused no vapour lock, additions of casing head gasoline were made until locking occurred at approximately 40° F. above atmospheric temperature when driving at 40 m.p.h., a speed which was selected because it gave maximum fuel system temperatures. To eliminate weathering effects samples were removed from the fuel systems at the point of vapour lock, *i.e.*, (1) vacuum tank outlet for vacuum types, (2) sediment bowl for gravity types, (3) generally the pump inlet for fuel pump types. Reid vapour pressures at 100° F. were determined on all these samples. The actual vapour lock temperatures were compared with those predicted from the equation: $t = 259 \cdot 140 \log. pr$, where t = vapour locking temp. in ° F, pr = Reid vapour pressure at 100° F.; and it was found that for fuel systems of (*a*) vacuum type, the agreement was good; (*b*) gravity type, the calculated temperatures are 4-5° F. lower; (*c*) fuel pump type, the relationship is variable but general agreement good.

A scale is presented which enables the vapour locking temperature of a gasoline to be read from the known Reid vapour pressure. The average observed vapour locking temperature was 33° F. (max. 48° F.) above atmospheric, so that for summer temperatures of 105° F. it is necessary to use a gasoline having a vapour pressure not exceeding 6·5 lb. Reid in order to ensure freedom from vapour lock in more than the average number of cars. It is therefore necessary for oil companies to extend the use of stabilising equipment and to remove excessive amounts of propane. Later models of some car manufacturers indicate that the automotive engineer is striving to lower fuel system temperatures in order to prevent vapour locking.

R. A. E.

861. Investigators Advance Methods for Eliminating Vapour Locking. O. C. Bridgeman. *Oil and Gas J.*, 1931, **29** (48), 122.

Forty-two samples of gasoline of widely different properties, including mixtures having maximum contents of 40% benzol, 20% butane and 5% propane have been examined by various laboratory methods and in laboratory fuel feed systems. Using the Bureau of Standards method for determining gas-free vapour pressures as a basis it has been found that:—

(1) For propane-free gasolines having conventional distillation curves, the temperature on the A.S.T.M. distillation curve at which 10% is evaporated represents essentially the maximum fuel temperature in the feed system

without encountering vapour lock. Trouble may be encoutered at lower temperatures in the case of gasolines containing propane.

(2) The liquid I.B.P. temperature on A.S.T.M. distillation is equal to the normal bubble point, but the method is inaccurate.

(3) Reid vapour pressures of 90° F. or 100° F. show satisfactory agreement with gas-free vapour pressures when multiplied by the factor 1·1. The relation between Reid vapour pressure in lb. per sq. in. (pR) at 100° F. and the vapour lock temperature (t) in ° F. at sea level can be expressed by the equation $t = 250 - 140 \log pR$. Vapour locking temperatures are reduced by approximately 2° F. for every 1000 ft. increase in altitude.

A chart is presented showing the flow in gal./hr. for varying fuel pump inlet temperatures and car speeds. Tables are also given to show the increase above atmospheric temperature at the critical point in 27 different automobiles driven at 40 m.p.h., idling after a 40 m.p.h. run and during the climb up a six mile mountain.

Since the majority of recent cars are equipped with fuel pump systems, only these systems are further considered. A chart is drawn with atmospheric temperature and Reid vapour pressure in lb./sq. in. as co-ordinates showing when vapour locking occurs in the worst, best and average type of car tested and an ideal line is plotted on the assumption that the fuel system temperature does not rise above atmospheric. As the temperatures relate to gasoline in the fuel system about 1 lb./sq. in. can be added to the vapour pressure to apply to products leaving the refinery. At 90° F. the ideal line requires a maximum vapour pressure of 16 lb.; the average and best cars tested maxima of 8 and 13·5 lb. respectively, showing the improvement that can be effected in design of fuel systems.

Fuel system temperatures can be reduced by the following simple changes: (1) Transferring the fuel line between rear tank and pump to the outside of frame channel; (2) insulating thermally the fuel feed line; (3) suitable location of fuel pump to reduce heat conduction from engine; (4) cooling and shielding the pump. R. A. E.

862. Vapour Lock: A Problem of Motor Design. R. C. Conine. *Oil and Gas J.*, 1931, **29** (49), 26.

At a recent meeting of the A.P.I. at St. Louis there was a discussion on Dr. Bridgeman's paper (Abstr. No. 861) on vapour lock. Dr. Wilson said that motorists should report vapour lock troubles, so that manufacturers would have to investigate rearrangement of fuel systems to eliminate the trouble. Dr. G. Oberfell presented data showing the increase in vapour pressure (Reid at 100° F.) caused by additions of 1% of propane, *iso*-butane and *n*-butane to motor fuel to be 2·43, 0·73 and 0·50 lb. respectively. He also showed that for atmospheric temperatures ranging from 100° F. to 60° F., the allowable percentages of "butanes" in the gasoline could be increased by 8·7/16·7% if the fuel system of the average car were improved to equal the standard of the best car tested. The octane number of *n*-butane is 91·5, of *iso*-butane 100, and butylenes probably have a higher anti-knock value. Improvement in fuel system design would thus conserve resources and permit an increase in anti-knock value and effective volatility of motor fuel by virtue of increased butane usage.

The increase in vapour pressure of the average motor fuel during the last ten years has been approximately 3 lb. (Reid at 100° F.). This is due to increased cracking, better conservation of vapours in crude and better recovery of volatile constituents from refinery gases. The percentage of natural gasoline to total motor fuel marketed in the U.S.A. was 10 in 1923 and 11·72 in 1930, so that the increase in vapour pressure has not been brought about by increased use of natural gasoline. R. A. E.

863. Motor Fuels. H. W. Elkington. E.P. 345,587, 26.3.31. Appl., 20.6.30.

The patent deals with the manufacture of anti-knock motor fuels comprising the addition of a benzine-soluble mixture of aromatic amines to a benzine motor fuel.

The aromatic amines are obtained by the direct nitration and reduction of crude hydrocarbon mixtures containing benzene and its homologues.

W. H. T.

864. Motor Fuel. F. J. Koblitz. E.P. 347,132, 22.4.31. Appl., 22.1.30.

The patent describes a fuel-improving composition for I.C. engines. The composition comprises: 15 parts of aniline, 3 of anthracene, 5 of camphor, 5 of castor oil, 10 of naphthalene, 12 of tetrahydronaphthalene, 6 of decahydronaphthalene, 8 of hexhydronaphthalene, 1 of nitronaphthalene, 1 of picric acid, 10 of sulphuric ether and 5 of cresol. In addition to the above the composition may also contain 5½ parts acetone, 6 of monochlor-naphthalene, ½ of acrolein, 2 of hydroquinone and 5 of benzoic acid. The proportions of the solid substances are in grammes and those of the liquids in c.c.

W. H. T.

865. Production of Non-Knocking Motor Spirit. D. A. Howes and the I.C.I., Ltd. E.P. 347,380, 30.4.31. Appl., 28.2.30.

Non-knocking motor spirit is produced by destructively hydrogenating carbonaceous material and blending the spirit fraction with that obtained by the pyrolysis of the waste gases. These are preferably freed from paraffins above ethane by scrubbing with a suitable solvent under pressure before pyrolysis. Any proportion of pyrolysis to hydrogenation spirit may be used, but a proportion of 1 : 54 is suggested. It is also suggested that the pyrolysis spirit should be cut at 185° C., and the hydrocarbon fraction at 150° C.

S. F. B.

866. Fractional Distillation of Natural Gasoline. F. S. Woidich. U.S.P. 1,803,581, 5.5.31. Appl., 6.11.24.

Apparatus for the stabilisation of natural gasoline, in which the permanent gases are removed and collected without the usual losses of natural gasoline, consists of a system for storing natural gasolines at different pressures, and a thermo-syphon heater above which is situated a rectifying column. A mixing head is attached near the base of the latter for conducting fluids through the heater, to and from the bottom of the column. An expansion chamber is connected to the top of the column and flues extend through the chamber to conduct gases and vapours from the column to heat the expanded gasolines in the chamber. A refrigerating chamber is mounted on top of the expansion chamber, and a pipe connects the top of the former to the mixing head, while a second pipe attached to the bottom of the refrigerating chamber is connected to the column at different levels. W. H. T.

See also Abstract No. 938.

Lubricants and Wax.

867. Repress to Make Zero Cold Test Oil. W. T. Ziegenhain. *Oil and Gas J.*, 1931, **29** (44), 83.

The Magnolia Petr. Co. is now making zero cold test neutrals from paraffinic mid-Continent crude without chilling to temperatures much below the cold test of the finished oil. This is effected by (1) a close correlation of the operation of the wax distillate stills with two simple laboratory tests and by (2) the installation of equipment for double pressing.

(1) It has been found that maximum yield of finished neutrals is indicated by high viscosity in the wax distillate, while ease and completeness of dewaxing is shown by the effectiveness of the fractionation of the wax distillate. For the mixture of Mid-Continent and Panhandle crude handled, the wax distillate is held at the optimum viscosity of 66 to 68 secs. at 100° F. A Hempel distillation at 10 mm. pressure on the wax distillate should give a nearly straight distillation curve. Also a minimum percentage should distil above 510° F. (at 10 mm. pressure at the top of the column).

Percentages over 7% and in general a sharp upward turn in the curve after 510° F. usually result in the formation of a gummy wax cake during pressing.

(2) The filtration plant is of conventional type but includes direct expansion chillers and is generally of improved design and very smooth in operation.

An essential step in the process is allowing the wax distillate to be held undisturbed in storage for 15 days after distillation to allow the wax crystals to grow to a size more convenient for filtration. The oil is then cooled in Gray chillers and pressed at a temperature of 30° F. A small percentage of removable wax is allowed to come through in order to build up a more porous cake in the second repressing to remove the softer wax. After the first pressing the oil is run to the direct expansion Carbondale chillers and cooled to—8° F., and then pumped to Carbondale presses. The wax formed in this operation is quite dry and easily removed from the blankets.

A flow sheet of the plant and a description of the Carbondale chillers and pumps employed is given. C. L. G.

868. Use of Olive Oils in Lubrication of I.C. Engines. N. Champsaur. *Ann. des Comb. Liquides*, 1930, **5**, 987–1007.

The use of olive oils is recommended for lubrication of I.C. engines, either alone or mixed with mineral oils. It is stated to be superior to mineral oils, having greater oiliness, forming less carbon and the viscosity falling off less with temperature. The addition of 5–10% olive oil to mineral oils tends to improve the latter considerably. The effect on carbon formation is due to the oxygen content of the oil, which tends to make combustion more complete. A discussion of the chemical and physical properties of olive oil is given and it is recommended that the acidity should not be over 4% as oleic acid and the congealing point about 5° C. Comparative viscosity curves of the oil with mineral oils of different types show the very flat nature of the curve with temperature.

Tests were carried out on tractors and automobile engines to compare the effects of the two classes of oil. About 3% greater output was obtained from the olive oil; this also facilitated starting. It is stated that olive oil is satisfactory for engines of up to 100 mm. bore, while for larger cylinders, heavy mineral oils containing up to 50% olive oil are suitable. E. B. E.

869. Chemical Composition of Lubricating Oils. M. Bestushew. *Erdöl und Teer*, 1931, **7**, 159–161, 191–193, 205–208.

The nature of the aromatic and naphthene hydrocarbons in lubricating oils has been investigated, especially in relation to the technical value of the oils. For the experimental work, oils from five distinct Russian fields were used, the oils being first fractionated and analysed. The fractions were then treated with butanone or ether-alcohol, to remove paraffin and with strong sulphuric acid to remove aromatics. The specific gravities, viscosities, elementary analysis, series, etc., are given for the fractions and for the various naphthenes and aromatics, in tabular form. As the boiling point and molecular weight increases the ratio of H : C becomes less, indicating more complex ring

structures. The early fractions of some oils (e.g., Balachany) consist of simple rings (e.g., cyclohexane and benzene derivatives); in the higher fractions naphthalene, anthracene and hydrogenated bi- and tri-cyclic compounds occur. Paraffin-base oils are distinguished by the simplicity of the ring structure of their constituents and asphaltic oils by their more complicated structures. These results are compared with those of Mabery and others, e.g., Pennsylvanian and Grosny oils show similar structures, while Californian and Louisiana oils resemble those from Kalushsky in being more complicated. In asphalt-base oils the amount of benzine is small as this consists of paraffin and simple-ring hydrocarbons, while the simplest polycyclic compounds boil about 200° C. Paraffin-base oils seem also to show more carbon atoms in the side-chains.

The viscosity of lubricating oils is thought to be chiefly due to polycyclic aromatic hydrocarbons, these having far higher viscosities than the naphthenes of the same boiling range, but steeper viscosity-temperature curves. Removal of aromatics by sulphuric acid always greatly decreases the viscosity. The effect of paraffin is to lower the viscosity and flatten the temperature curve. The polycyclic aromatic and naphthene compounds show less resistance to heat and oxidation as the boiling point rises and the structure becomes more complicated. The results of oxidation with nitric acid are given. Heavy fractions from Kalushsky oils are very readily attacked while simple-ring compounds are fairly stable.

Results of Conradson tests also show that coke formation is chiefly due to complicated aromatic hydrocarbons. The iodine numbers of these high fractions are thought to be due to substitution in aromatic nuclei.

E. B. E.

870. Properties and Structures of Paraffin Waxes and Ceresines. A. N. Sachanin, L. G. Zhedeva and A. N. Vasilyev. *Nat Petr. News*, 1931, **23** (16), 49; (17), 67; (18), 51; (19), 71.

Both ceresines and paraffins comprise homologous series of hydrocarbons of the paraffin series. Normal paraffins and so-called iso or amorphous paraffin obey the same law of solubility of crystalline substances and differ only in their crystal structure.

Ceresines, but not paraffins, form stable mixtures with oil. Hence ceresines or paraffins containing ceresines cannot be freed from oil by sweating. Ceresine can be distilled unchanged at pressure of 0·0003 mm. Hg. and temperature of 330° C., but at higher pressures decomposition to paraffin occurs. Paraffin wax in a distillate is not a decomposition product entirely, but exists as such in the crude and distils over with the appropriate oil fraction. Ceresine is less stable chemically than paraffin and is easily attacked by fuming sulphuric acid.

Ceresines have higher specific gravity, molecular weight, viscosity, b. pt. and nitrobenzine point than a paraffin with the same m. pt. Pure ceresine and pure paraffin may be identified by their crystal structures, ceresine crystallising in needles and paraffin in the form of strips. The crystal structure of each is very much modified by the admixture of the other. H. G.

871. Carnauba Wax Bleaching. *Chem Trade J.*, 1931, **88**, 486.

H. Nast (*Seifensieder Z.*, Jan. 22nd, 1931) states that Carnauba wax can be bleached to a pale yellow colour by the use of a strong oxidising agent in the presence of certain solvents or emulsification agents which probably also act to some extent as oxygen carriers. Chromic acid, peroxides, peracids and persalts are mentioned as satisfactory oxidising agents and organic acids and their esters as effective media for the reaction. R. A. E.

872. Lubricating Compositions. Panoleum Co. E.P. 347,574, 27.4.31. Appl., 25.10.29.

A lubricating composition consists of 80–90% by wt. of lubricating oil, 5–15% of an amide or anilide of a higher fatty acid, 3–5% of a metallic soap. The amide or anilide, e.g., stearanilides, have the property of stiffening the composition to a high degree compared to its proportion in the composition. The metallic soap raises the m. pt. of the composition considerably. The oil and soaps must be substantially free from water. W. D. S.

873. Purification of Lubricating Oils. F. X. Govers and Indian Refining Co. U.S.P. 1,802,942, 28.4.31. Appl., 18.10.28.

The manufacture of a low cold test lubricating oil from a wax distillate fraction of a mineral oil which is substantially free from constituents having a b. pt. equal to or below that of gas oil, is described. The fraction is mixed with acetone and benzol in such a ratio that the acetone and benzol have complete solvent action on the fraction at 100° F., and complete solvent action on the liquid hydrocarbons but not on the solid hydrocarbons at —5° F. Thus the solid hydrocarbons can be removed and the solvents recovered. The resulting oil has a cold test approximately the same as the temperature at the time of the removal of the solid hydrocarbons. By cooling the oil to 0° F. or below, further hydrocarbons may be precipitated.
W. D. S.

874. Production of Lubricating Oils. E. R. Lederer and Texas Pacific Coal and Oil Co. U.S.P. 1,803,941, 5.5.31. Appl., 5.3.29.

A method for producing fully dewaxed, unblended paraffin-base lubricating oil of a cold test of approximately 0° F. is described. The starting material is a paraffin-base crude oil from which the lighter fractions have been distilled to produce a still-residue containing wax substantially all of which is an amorphous form. This residue after ageing is treated with sulphuric acid (35 lb./brl. of 98%) below 125° F., stored to remove the acid sludge and then treated with filter clay at 420° F., i.e., below the temperature which would cause conversion of amorphous to crystalline wax. The temperature is reduced to 135° F., the clay-treated oil diluted with naphtha and the clay filtered off. The oil is further diluted and chilled at a rate approximately 2½°/hr. to about 10° F. and more rapidly to —20° to —40° below zero. The wax is then centrifuged from the oil and the diluent separated from the dewaxed oil. This oil has a cold test of from zero to —15° F., a f. pt. above 480° F., a Saybolt viscosity of from 88″ at 210° F. to 155″ at 210° F., and free to flow at pump pressures of 7 to 10 lb. as low as —40° F. W. D. S.

See also Abstract No. 890.

Fuel Oil.

875. Fuel Oil Burners. Lynn Products Co. and L. A. Popp. E.P. 345,578, 26.3.31. Appl., 6.6.30. Conv. (U.S.A.), 3.3.30.

The burner in its preferred form has a number of annular, concentric combustion chambers, the main object being to provide for the delivery of the right proportion of combustible vapour to each combustion chamber so as to produce a balanced heat distribution and thus increase the capacity of the burner. A central vapourising chamber is provided with a number of conduits for delivering vapour to the combustion chambers. Some conduits deliver to both and others to only one combustion chamber. R. W. L. C.

876. Purifying Hydrocarbons. J. F. A. Bruzac. E.P. 346,152, 9.4.31. Appl., 2.4.30.

Crude hydrocarbons intended for fuel in I.C. engines are purified by treatment with oxygen, ozonised air or ozonised oxygen followed by washing with an alkaline lye. S. F. B.

877. Purifying Liquid Hydrocarbons. J. F. A. Bruzac. E.P. 436,153, 9.4.31. Appl., 2.4.30.

Heavy hydrocarbons are treated either by (1) heating at a temperature, not exceeding 500° C. in contact with non-reacting metals such as iron, copper, nickel or the like, or with pebbles under a pressure equal to or slightly above atmospheric and condensing the resulting vapours; or (2) leading the vapours from them obtained by distillation at a temperature not exceeding 500° C. into a receiver at the pressure given above containing non-reacting material and condensing the resulting vapours. The distillates obtained by either method are treated with oxygen, ozonised air or ozonised oxygen to remove carbon and sulphur and to purify and deodorise them. S. F. B.

See also Abstract No. 916.

Asphalt and Fluxes.

878. Bitumens and Their Use in Paints. W. N. Bowran. *Oil and Colour Trades J.*, 1931, **79**, 1171–1175.

Resistance to weathering was found to be the greatest for asphaltites (Gilsonite, Manjak or glance pitch, and Grahamite), the native asphalts (Bermudez) being inferior. Blown asphalts were worst and were found to be not entirely satisfactory for under-water purposes. Bitumens, however, are now acknowledged to be among the most weather-resistant substances known for use in the paint trade.

In some water absorption tests, the thickness of film was found to have an influence on the amount of absorption. Also, the replacement of gum in carriage varnish by bitumen caused the carriage varnish absorption of 10·3% in the first three days to drop to 1·9%. Black japans gave surprisingly good results, the larger the percentage of bitumen present the lesser the absorption.

The elasticity of bitumens has a bearing on their suitability. Blown or residual bitumens are elastic to a degree and are admirable for roofing compositions. Native asphalts and asphaltites are too hard to be used without the incorporation of linseed and/or tung oils which impart elasticity. Fatty acid pitches are used in air drying compositions, whilst mineral oils serve the same purpose in stoving varnishes. Blown bitumens are particularly useful for paints subject to vibration or extreme cold (refrigerators) and because of their adhesiveness, elasticity and low susceptibility factor.

Several other conclusions arrived at were: Bitumens alone in solution are deficient in holding pigments in suspension, neither can they alone yield a well-bound film. Bitumens also retard the oxidation of the oil incorporated. Residual asphalts are the worst offenders and the native asphalts come next.

The most suitable driers for low bitumen mediums at present are lead and cobalt driers. Aromatic hydrocarbon thinners whilst superior in solvent power to white spirit are unfavourable because of their physiological action,

and petroleum solvent naphtha (120° C.–160° C.) is also useful on account of its "keenness" in solvent action. In general, the softer the bitumen used, the lighter (in fraction) should be the solvent.

Bitumen paints in their application to iron and steel surfaces depend for their value on at least three properties: (1) they are neutral and do not create or support electrolyte action; (2) they adhere with great tenacity to metal surfaces; (3) they are impervious to water. Paints of high bitumen content are not recommended for application to wood. C. I. K.

879. Cold Tar Road Coverings. D. J. Pickée. E.P. 346,025, 30.3.31. Appl., 30.12.29. Conv. (Germany), 23.8.29.

The invention relates to an improved process for the production of a cold asphalt street covering comprising a mixture of tarry substances with or without other carbonaceous materials, and with bituminous materials and hydrated lime. The latter is admixed in the proportion of 10–12% of the weight of the end product and is added either dry or with the addition of water as milk of lime. A suggested mixture contains 100 parts by weight of tar, 25 parts of asphalt and 40 parts of milk of lime. A solvent such as benzene or benzene-water mixture may be added as a diluent to the mixture, while in certain cases powdered lime may be utilised, the addition of water being effected later during the building of the road. W. H. T.

880. Preparation of Tarry and/or Bituminous Solutions. R. Arnot. E.P. 346,681, 15.4.31. Appl., 15.10.29.

The patent comprises the addition to a mass of tar, bitumen or bituminous material, which naturally may contain unsaturated compounds, of a flux medium or diluent. This may be in the form of liquid or oil containing unsaturated hydrocarbons with which the unsaturated compounds of the preponderating mass will resinify, condense, and/or polymerise under the influence of air or oxygen. W. H. T.

See also Abstracts Nos. 849, 914, 939.

Special Products.

881. Manufacture of Conversion Products of Higher Paraffin Hydrocarbons. A. Carpmael. E.P. 343,948, 11.2.31. Appl., 11.9.29.

The halogenation products of liquid and solid paraffins derived from petroleum or brown coal containing more than 8 carbon atoms and at least 2 halogen atoms in the hydrocarbon mol., are treated in an aqueous medium under pressure with caustic alkalies or alkali carbonates except bicarbonates or alkaline earths. The products obtained are unsaturated viscous light-coloured oils which are used as lubricating or greasing agents. W. S. E. C.

882. Conversion of Methane to Liquid Hydrocarbons by Thermal Treatment. N. V. Silica en Ovenbouw Mij and C. Otto. E.P. 344,443, 27.2.31. Appl., 27.11.29.

The methane is led into passages provided within the brickwork of a coke oven and the walls of the passages are heated by contact with the gaseous products of combustion burnt in other passages within the brickwork. Details of such an arrangement are given. It is claimed that 1040 to 1060° C. is the best temperature range for the conversion of relatively pure methane. If required steam may be introduced with the methane. E. N. H.

883. Production of 1.1.2-Trichlorethane. I. G. Farbenind. A.-G. E.P. 344,592, 12.3.31. Appl., 10.1.30.

A mixture containing acetylene, chlorine and hydrogen chloride is caused to react by means of contact materials such as coke, chamotte, quartz, clay, rock salt, active carbon, silica or pumice impregnated or not with metallic salts. The operation is carried out in a cycle, the reaction taking place in narrow or constricted spaces or in the presence of excess acetylene or hydrogen chloride or inert foreign gases in amounts sufficient to prevent explosions. The contact materials are either heated or else the mixture is heated alone.
W. S. E. C.

884. Removal of Tar Acids from Oils. South Metropolitan Gas Co. and H. Stanier. E.P. 344,614, 12.3.31. Appl., 31.1.30.

A process is described of removing tar acids from hydrocarbon oils by extraction with an organic base which is substantially insoluble in the oil. The base is obtained by the interaction of an alkyline oxide and ammonia or in the form of hydrochloride by interaction of an alkyline mono-chlorhydrin with ammonia. The tar acids may be separated from the organic base (triethanolamine) by extracting the mixture with water with or without saturation of the water with acidic gas. Any neutral oil which may have been extracted together with the tar acids may be removed either by extraction with a petroleum oil or by distillation.
W. H. T.

885. Purification of Synthetic Alcohols. J. R. Park and I. C. I., Ltd. E.P. 344,796, 10.3.31. Appl., 10.12.29.

Synthetic alcohols prepared by catalytic hydrogenation of oxides of carbon under pressure are often contaminated with iron compounds, e.g., iron carbonyl, which cannot be completely removed by simple distillation. These iron compounds are removed from the alcohol by prolonged treatment with alkali. The operation is carried out by allowing the impure alcohol to stand for several days at ordinary or raised temperatures with solid caustic soda or other alkaline substance. During this treatment the mixture may be stirred by a slow current of air, and this is probably an advantage, as the presence of dissolved oxygen in the liquid is believed to play a part in the purification. After filtration (if necessary) the alcohol is distilled off.
W. D. S.

886. Manufacture of Sulphonated Conversion Products of Higher Paraffin Hydrocarbons. A. Carpmael. E.P. 344,829, 11.3.31. Appl., 11.9.29.

The products obtained by the action of an aqueous or alcoholic solution or suspension of an alkali carbonate or alkaline earth on halogenated derivatives of paraffin hydrocarbons containing more than 8 carbon atoms and at least 2 halogen atoms in the hydrocarbon molecule are treated with a sulphonating agent. Sulphuric acid monohydrate, fuming sulphuric acid or other sulphonating agent is used with or without the addition of a suitable diluent such as carbon tetrachloride, trichlorethylene, petroleum ether or of compounds assisting the sulphonation, such as lower fatty acids or their anhydrides or chlorides.
W. S. E. C.

887. Manufacture of Butadiene or Its Homologues. I. G. Farbenind. A.-G. E.P. 345,270, 18.3.31. Appl., 18.12.29.

Butadiene or its homologues is obtained by passing the vapour of the corresponding unsaturated alcohol, having 4 or more carbon atoms in its molecule, at a temperature above 200° C., either alone or diluted with water over a catalyst which has a dehydrating action, e.g., sulphates, particularly alum or a mixture of alum, acid phosphates, etc.
S. F. B.

888. Manufacture and Production of Oxygenated Organic Compounds. I. G. Farbenind. A.-G. E.P. 345,391, 26.3.31. Appl., 16.1.30. See also E.P. 312,388.

Liquid hydrocarbons of the benzine series, *e.g.*, petroleum fractions, boiling from 40° C. to about 130° C., are converted into oxygenated compounds by oxidation in the liquid phase at elevated temperatures, while avoiding distillation, by means of a gaseous oxidising agent, if desired at elevated pressures, in the presence of certain metallic catalysts, the course of the reaction being influenced to give alcohols or esters by adding a carboxylic acid to the hydrocarbon as diluent. The oxidising agent employed may consist of oxygen, air, nitrogen oxides or mixtures of any of these, while organic compounds of the alkali or alkaline earth metals or of magnesium, aluminium or manganese such as the resinates, oleates or enolates may be used as catalysts. The carboxylic acid used as diluent should form at least 2% of the hydrocarbon used and should be one of the lower saturated aliphatic monocarboxylic acids containing up to 4 carbon atoms; the corresponding anhydride may be used in place of the acid. Examples are given of the oxidation of hexane and light petroleum fractions to the lower alcohols and their esters, using manganese acetyl acetonate as catalyst and air as oxidising agent. S. F. B.

889. Heavy Oil in the Manufacture of Carburetted Water Gas. Humphreys and Glasgow, Ltd., assrs. of J. A. Perry. E.P. 345,577, 26.3.31. Appl., 6.6.30. Conv. (U.S.A.), 27.7.29.

A shallow fuel bed is situated at the bottom of a carburetting chamber, the latter being unobstructed by heat absorbing material such as checkerbrick. The generator fuel bed is steamed and the water gas produced passed through the carburetting chamber from the top towards the shallow fuel bed. Simultaneously, heavy oil is sprayed into the top of the carburetting chamber so that the resultant coke is deposited on the fuel bed. The carburetted gas is led off from the chamber above the fuel bed and the deposited coke subsequently consumed. W. H. T.

Refining and Refinery Plant.

890. Atmospheric and Vacuum Distillation. J. Primrose. *Oil and Gas J.*, 1931, **29** (43), 20.

Modern refinery practice in the production of overhead cylinder stock is discussed with reference to the advantage of a vacuum stage in the distillation, as illustrated by the results of runs on Seminole and Mid-Continent crude.

Single flash distillation to heavy flux in an atmospheric unit has the disadvantage that at the high temperature required to vaporise the heavy cylinder oil fractions, the colour stability of the benzine and kerosine is impaired. By using, however, a secondary flash chamber, a satisfactory yield of cylinder stock can be obtained at temperatures not harmful to the colour of the lighter oils. A flow sheet of a typical modern two stage atmospheric and vacuum unit is given with a full description of operating conditions, most suitable type of control, fractionating and heat exchanging equipment.

A table is given showing the yields from Seminole crude in an atmospheric unit and an indication given of the improved results given by the addition of a flash chamber. Curves are given showing the distillation ranges and

colours of the gasoline and kerosine streams when running with various still temperatures. At 750° F. the gasoline was +30 and the kerosine 24, while at 800° F. the gasoline was 22 and the kerosine 17/18 Saybolt. Further comparative yields are given from Seminole and Mid-Continent crude through a two stage atmospheric and vacuum unit (*a*) using an auxiliary tower and (*b*) without the auxiliary tower.

The increase in the yield of cylinder oil is small in this case, although the colour of the lighter streams is improved, so that installation of an auxiliary flash tower would depend on economic considerations.

The flexibility of the type of plant described is shown by the yields from various crudes, products of widely varying grade being obtained. C. L. G.

891. Discuss Pressure Distillate Stabilisation. A. W. Burket. *Oil and Gas J.*, 1931, **29** (43), 34.

Preventable refinery losses can often be traced to the lack of an adequate water cooling system. In general the circulating water should be cooled to within 5° or 10° F. of the atmospheric wet bulb temperature. Higher temperatures lower the efficiency and increase the cost of vapour recovery systems, though they are often resorted to in topping plants in order to produce a fairly stable product without actual stabilisation. This is not so effective in the case of the streams from a cracking plant, owing to the much larger amounts of propane present and to the higher condensing pressures. This propane can only be removed effectively and economically by stabilisation. It should be remembered, however, that each volume of propane removed from the pressure distillate can be replaced with 3½ vol. butane without altering the final vapour pressure. Modern vapour recovery plants are therefore designed to recover 90% of butane, though this high recovery is not profitable, unless the butane is sold separately or can be mixed with propane free distillates. An important factor in the design of fractionating equipment is the maximum temperature, which should be below that at which the gums present in cracked distillates are liable to polymerise.

A basic flow diagram for a fractionating plant is given, and the various factors influencing the design of the equipment are fully discussed.

C. L. G.

892. Minimising Corrosion in Condensers. A. E. Pew. *Oil and Gas J.*, 1931, **29** (48), 112.

Corrosion of condensers on the water side may be due either to (*a*) mechanically suspended matter, such as mud, sand, etc., or (*b*) soluble salts in the cooling water, particularly if sea water is used. On the oil side of the condenser, corrosion is usually caused by (*a*) sulphur and its gaseous derivatives, or (*b*) salts and their acid derivatives in the water associated with the crude oil.

In the case of corrosion by water, it does not appear at first sight to be practical to treat the large quantities of water necessary for cooling. If, however, cooling towers are used, the condensed water can be recirculated, only sufficient water for make up (usually 5%) being added subsequently. Treatment of this water then results in considerable economies of equipment, water requirements, elimination of corrosion, etc. The *p*H value of treated water should be maintained at 10 or higher to reduce tendency to corrosion. Much of the corrosion trouble is due to electrolytic action between dissimilar metals, particularly in the presence of dissolved oxygen.

In the case of corrosion on the oil side, hydrochloric and sulphuric acid are the two main corroding agents. The former chiefly originates from magnesium chloride, and is usually combated by the introduction of anhydrous

ammonia or caustic soda. The former is, however, rather expensive, while also there is danger of plugging due to ammonium salts. Caustic soda is frequently successful, but if used in large quantities affects the ash content of the fuel oil, with deleterious results owing to the subsequent scale deposition in boilers and on firebrick, etc. Hydrogen sulphide is best removed by hydrated lime and by the circulation of caustic soda solutions through the condenser. The use of triethanolamine as an absorbent has also been suggested, this material being non-corrosive, non-inflammable, able to absorb 100 vols. of H_2S, and easily recovered by heating to 212° F.

Indications are given of the types of alloy found most suitable for condensers for different types of crudes.

Attention is drawn to the advantages of removal of water from crude by electrolytic dehydration instead of distillation, the salts being also removed.

Tables are given showing the chloride content of various typical crudes, with descriptions of methods of determining the chloride content. Figures are also given of the conventional shell and tube type of condenser, and a preferred design in which there are no shell surfaces in a horizontal position and no baffles, while the bottom head may be protected with cement, etc.

C. L. G.

893. Run De Florez Cracking Units to Coke. P. F. Swanson. *Oil and Gas J.*, 1931, **29** (42), 95.

In cracking oil to coke the quality of the coke must be controlled, the process made continuous and operating costs reduced. In a De Florez plant charging stock is admitted to a cracking tower in which it meets vapours from the heater. The vapours pass to a fractionating tower, the bottom of which forms an accumulator tank for the cycle stock. The vapours from the fractionating tower are condensed, and the gasoline recovered under a pressure of about 60 lb. in a separator. Part of the gasoline is circulated to the fractionating tower, and the remainder passed to a weathering drum where the pressure is reduced to several inches of vacuum. The gases from separator and weathering drum are passed to an absorber, in which cooled cycle stock is used as absorbing medium, the gases pass overhead and the enriched oil is returned to the towers as reflux.

The cycle stock from the fractionating tower is passed to a small economiser, thence to the vertical heater tubes and to the transfer line leading to 2 coke drums. Into this transfer line residuum from the cracking tower is injected by pump, so that the products entering the coke drums are virtually all vaporized and pass overhead to the cracking tower, thus completing the cycle. The quality of the coke is controlled by the quantity of residue pumped and the temperature of the drums. The cracking tower has several bubble trays, and since only distillate passes to the accumulator section of the fractionating tower, practically no carbonization takes place in the tubes. The coke drums are vertically disposed and provided with a drill pit and bit for cleaning, and are used alternately, so that whilst one is in operation the other can be cleaned. It is possible to operate at temperatures ranging from 1000° to 1070° F., according to the nature of the stock and the products required, and to regulate the percentage of volatile matter in the coke between 3 and 12. The coke drums are filled to ⅔ of their height before switching over and require 12-13 hours to clean.

With the higher operating temperature the fuel consumption and gasoline yield are reduced, but the gas yield and anti-knock value of the gasoline increases. Photographs and a flow chart are provided.

R. A. E.

894. Refining Petroleum in Italy. G. B. Antonelli. *L'Ingegnere*, 1931, **5**, 178–186.

The paper opens with an outline of the treatment of crude petroleum to obtain the various commercial products.

The influence of taxation on the refining of petroleum in Italy is discussed at some length. The production of benzine by cracking is briefly noted, while hydrogenation is dealt with rather fully. The problem of refining in Italy is quite different from refining on the field, as the starting material has to be bought in the most economical markets and may, therefore, differ from time to time. This may call for considerable variation in the method of treatment. The most advantageous application of hydrogenation in Italy would be to use as starting material, a residue which is liable to the lowest rate of duty. This would be subjected to two successive operations: (1) to obtain gas oil and petrol; (2) to transform the gas oil to petrol. Comparing the relative merits of cracking and hydrogenating a gas oil, the latter process gives the higher proportion of petrol, but this advantage is somewhat compensated by the secondary products obtained by the cracking process. These latter may be valuable in the development of other industries. The production of lubricants by hydrogenation is not, at present, of much interest to the growing Italian petroleum industry.

In an addendum, distillation curves are given for straight gasolines, kerosines and gas oils from various sources, together with the boiling points of hydrocarbons from pentane to heptadecane and from amylene to tridecylene. In a second diagram, similar curves are given showing the variation obtained with benzines from different crudes. A third diagram gives the distillation curves for various special benzines as sold in France. C. C.

895. Distillation of Hydrocarbons. Barrett Co. (U.S.A.). E.P. 345,272, 18.3.31. Appl., 18.12.29. Conv. (U.S.A.), 18.12.28.

A still is described in which a rotating cylinder, situate at the base of the still, serves to pump upwards a spray of hydrocarbon. An inlet immediately above this allows hot gases to enter this spray, and the vapours so formed are led away to a settling tower, and thence to a condenser. Excess liquid hydrocarbon is returned via the curved top of the still. Coke formation, it is claimed, is reduced to a minimum. H. C. R.

896. Dephlegmator or Bubble Towers. H. Moore and J. B. Johnson & Co. E.P. 345,565, 26.3.31. Appl., 19.5.30.

The design of the dephlegmating columns or bubble towers is such that the liquid passing over one tray shall take as long a path as possible before passing to the tray next below it, thus facilitating intimate contact between the gases or vapours and the liquid. The desired effect is obtained by providing each of the trays, which are placed at spaced intervals in the height of the tower, with radial liquid inlet and outlet passages separated by a baffle to compel the liquid to make a circuit of the tower before descending to the next tray. A number of gas inlets surmounted by bubble caps are arranged in circular series on each tray, and the central bubble cap is provided with four extensions at right angles so arranged that the passage of liquid directly across the tray is prevented. R. W. L. C.

897. Refining Hydrocarbons. N. V. de Bataafsche Petr. Mij. E.P. 345,596, 26.3.31. Appl., 7.7.30. Conv. (U.S.A.), 15.7.29.

Sulphur compounds are removed from hydrocarbon oils by treating the oil with a dilute acid at atmospheric temperature. In a second stage, concentrated acid is used at a temperature substantially below atmospheric, while the third stage comprises another treatment with concentrated acid at a higher temperature (e.g., atmospheric). W. H. T.

898. Distillation of Petroleum Oils. Barrett Co., assrs. of A. A. McCubbin and J. Zavertnik. E.P. 346,407, 30.3.31. Appl., 30.9.29.

Petroleum oil is subjected to distillation using a pipe coil still in which it is heated to a temperature sufficient to vaporise a considerable proportion of the oil. The heated product is passed into a vapour separating chamber at approximately atmospheric pressure. The residual oil is drawn off while still at a high temperature to a high vacuum still in which practically all the oil which can be distilled at that temperature is vaporised. Lubricating oil stock may be used as charging stock. A description of a convenient plant is given. E. N. H.

899. Conversion of Hydrocarbons into Those of Higher Boiling Point. J. Y. Johnson and I. G. Farbenind A.-G. B.P. 347,727, 4.5.31. Appl., 2.1.30.

Hydrocarbons of high b.pt. (200°–350° C.) are treated with chlorine or other halogen, and then subjected to the action of activated aluminium (aluminium immersed in a solution of a salt of a metal which is less electro-positive) at 150°–250° C. The hydrogen halide formed during halogenation may be passed with the chlorinated hydrocarbons over the aluminium. The chlorinated hydrocarbons may also be mixed with unhalogenated hydro-carbon. Products with 60% boiling up to 200° have been obtained. Examples and diagrams are given. W. D. S.

900. Refining Hydrocarbons. E. C. Herthel, assr. to Sinclair Refining Co. U.S.P. 1,795,124, 3.3.31 Appl., 14.9.28.

Gasoline is supplied to the refining operation as a vapour mixture, including the addition of vapours of higher-boiling hydrocarbons in proportions such that the vapour mixture when condensed forms a condensate containing not less than about 50% and not more than about 75% of the gasoline product. The mixture is first passed through a condensing operation, whereby a part of the higher boiling hydrocarbons are condensed. The vapour mixture and condensate is passed through an adsorptive catalyst, and after-wards those constituents boiling higher than is suitable as components of the gasoline product are condensed. The remaining vapours are finally condensed to form the refined gasoline product. W. H. T.

901. Converting Residues Obtained on Sweetening Petroleum Distillates. H. G. M. Fischer, Assr. to Standard Oil Dev. Co. U.S.P. 1,795,278, 3.3.31. Appl., 25.8.27.

Sour petroleum distillates are sweetened by contact with added sulphur and a catalyst followed by redistillation under reduced pressure. The residue so obtained contains the added sulphur, together with much of that originally present in the sour distillate.

It is claimed that this oil may be converted to one of much lower sulphur content by a heat treatment. To this end the oil is heated in a pipe still to 500°–600° F. under a pressure of 5–50 lb. sq. in., according to whether vapour or liquid phase treatment is desired.

The heated oil is discharged into a separator from which the vapour consists chiefly of hydrogen sulphide and hydrocarbon sulphur compounds of lower b.pt. than those originally present in the residue. The oil obtained from the separator is characterised by a decreased sulphur content. H. B. T.

902. Refining Hydrocarbon Oils. A. S. Ramage, assr. to Gyro Process Co. U.S.P. 1,796,621, 17.3.31. Appl., 27.8.26.

Sulphur and chlorine are removed from hydrocarbons soluble in alcohol, by dissolving the hydrocarbons in an alcoholic solution of an alkali metal hydroxide, allowing the solution to stratify and separately removing the purified oil and the alcoholic alkaline solution containing the impurities. W. H. T.

903. Refining Hydrocarbons. F. A. Apgar, assr. to Sinclair Refining Co. U.S.P. 1,797,255, 24.3.31. Appl., 14.9.28.

Vapour phase refining is improved by passing the vapours successively through two separate bodies of the catalyst. Before complete deactivation of the first body, the sequence in which the vapours pass through the two bodies of catalyst is periodically reversed. W. H. T.

904. Refining Hydrocarbons. E. C. Herthel, assr. to Sinclair Refining Co. U.S.P. 1,797,262, 24.3.31. Appl., 14.9.28.

Improvements are described dealing with the vapour phase refining of cracked gasoline with an adsorptive catalyst. After passage of the vapours through the catalyst, high boiling polymers are separated and heavy constituents of the gasoline are fractionally condensed. The mixture of these two materials is subjected to vaporisation whereby oil components free from residual tar are obtained. The oil condensate is passed through the adsorptive catalyst with the hydrocarbon vapours to be refined as a liquid washing agent. W. H. T.

905. Method of Refining. R. C. Osterstrom and R. T. Tucker, assrs. to Pure Oil Co. U.S.P. 1,797,513, 24.3.31. Appl., 2.2.28.

This invention is particularly applicable to the refining of cracked motor fuels and concerns the removal of gum forming constituents. This is effected by adding an adsorbent (e.g., fuller's earth) to a flowing stream of the oil whereby gum forming constituents are polymerised. The mixture is subjected to super-atmospheric pressure and temperature in order to impregnate the adsorbent, after which the pressure is released and the mixture discharged into a vaporising and fractionating zone. Oil vapours and adsorbent, together with gumming constituents are finally discharged separately from the fractionating zone. W. H. T.

906. Treating Petroleum Oils. P. M. Travis. U.S.P. 1,798,341, 31.3.31. Appl., 13.7.27.

The invention relates to improvements in processes for the removal of suspended material from petroleum oils and residues, particularly residual oils from cracking operations. It is continuous in operation, and consists in passing the oil to be clarified through a horizontal solid discharge centrifuge at 120°–135° F. The centrifuge consists of an outer bowl and an internal helix shaped to the form of the bowl. The bowl and helix are rotated at high speed in the same direction, but a differential gearing enables the bowl to be rotated at a slightly greater speed than the helix, so that centrifugal separated solid material is continuously thrust along and discharged from the bowl while the clarified oil is discharged at another point. R. W. L. C.

907. Fractionating Liquids such as Mineral Oils and the Like. A. R. Earl and S. W. Reeves. U.S.P. 1,799,414, 7.4.31. Appl., 25.2.27.

A series of interconnected units are maintained under progressively varying pressures, a portion being above atmospheric, an intermediate section at atmospheric and the remainder below atmospheric pressure.

The oil is introduced into the series by way of the portion under sub-atmospheric pressure. Superheated steam is admitted to the portion under super-atmospheric pressure and flows through the unit, being allowed to expand in the sub-atmospheric portion.

The pressures of the steam in the different units is used for maintaining levels of the oil at variant altitudes in the unit system. These levels are relatively proportional to the pressures in the respective units while maintaining a quiescent flow of oil through a portion of the system.

Certain component fractions are removed from the unit under the lowest pressure. W. L.

908. Refining and Purification of Hydrocarbon Oils. J. C. Morrell, U.S.P. 1,799,431, 7.4.31. Appl., 25.3.26.

A process is described for the refining and purification of hydrocarbon oils of any gravity from a crude hydrocarbon oil of low gravity, up to and including a motor fuel of higher gravity; the method is particularly applicable to the desulphurising of cracked distillates. The oil is heated in the presence of added metal—e.g., zinc, tin or iron and glacial acetic acid. The refractory sulphur compounds are converted into hydrogen sulphide and mercaptans which can be subsequently treated so as to reduce the total sulphur content of the resulting product. W. S. E. C.

909. Refining Oil. A. E. Pew, assr. to Sun Oil Co. U.S.P. 1,799,640, 7.4.31. Appl., 14.6.27.

The invention relates to the process of distilling oil by means of mercury vapour, which is brought into physical contact with the liquid oil to be vaporised. The mercury vapour is at a higher temperature than the oil, the relative temperatures being such that the oil will condense the mercury vapour, the latent heat absorbed being sufficient to effect the vaporisation of a substantial proportion of the oil. The mercury is finally separated from the distillation residue. W. H. T.

910. Purification of Tar Acid-Bearing Oils. S. Caplan, assr. to Combustion Utilities Corpn. U.S.P. 1,799,780, 7.4.31. Appl., 25.9.26.

The invention relates to the purification of tar acid-bearing oils, particularly to a process for eliminating colour producing compounds. The oils are distilled and the vapours passed through a reflux column containing a porous filling impregnated or coated with a non-acidic ferric compound. The oil may also be mixed with a soluble ferric compound before distillation. W. H. T.

911. Distillation Process. S. P. Miller, assr. to Barrett & Co. U.S.P. 1,800,224, 14.4.31. Appl., 12.3.25.

In the distillation of tar in a shell still, a portion of the vapours from the vapour dome of the still are continuously withdrawn and returned to the still at the bottom of the tar, thus maintaining the tar in a state of reasonably violent agitation. The remainder of the vapours are fractionated, the refluxed condensate being returned to the still for redistillation. It is claimed that priming during the removal of water is obviated, that the yield of distillate oil is increased, and that coking of the residue is minimised. W. L.

912. Refining Mineral Oils. M. Teplitz, assr. to Corona Conversion Corpn. U.S.P. 1,801,213, 14.4.31. Appl., 18.11.25.

Oils containing substantial quantities of gum forming unsaturated compounds are first chlorinated by any suitable reagent such as free chlorine or a compound yielding chlorine in an available form. The chlorinated oil is

then heated and subjected to the action of a de-chlorinating reagent. This reaction may take place in either the liquid or vapour phase or both. A convenient method is to distill the chlorinated oil and to pass the evolved vapours upwards through a tower packed with zinc turnings.

It is usually advisable to soda wash the final distillate to remove traces of free chlorine.

During the distillation preheated natural gas may be passed into the still. The nascent unsaturated hydrocarbons produced in the zinc tower react with the methane and other constituents of the natural gas to form saturated compounds. W. L.

913. Treating and Rectifying Vapours. P. J. Carlisle, assr. to Roessler and Hasslacher Chem. Co. U.S.P. 1,801,412, 21.4.31. Appl., 25.11.27.

Vapours undergoing rectification in an ordinary column are subjected to the action of an alkali metal, usually sodium suspended in the reflux liquid. The alkali may be introduced as a spray of molten metal, as a colloidal suspension, as a finely divided solid metal or in any other suitable form. A method suggested consists of passing a condensed electric spark between two sodium electrodes submerged in a liquid such as ether, xylene, etc. The resulting colloidal suspension is introduced directly into the top of the column. The method preferred is to agitate condensate from the overhead of the column with metallic sodium at a temperature above the melting point of the latter, and feed the resulting suspension to the column at such a rate that substantially all the alkali is utilised in the upper part of the column. W. L.

Cracking.

914. Obtaining Asphalt by Cracking. G. Egloff and J. C. Morell. *Mon. Petr. Roumain*, 1931, **31**, 483–484.

Cracking can be adapted to produce gas, petrol and asphaltic bodies, the quality of the latter depending on the technique adopted. It is usually considered that asphalts are formed by oxidation, sulphuration, polymerisation and/or condensation. A possible theory is that acidic polycyclic compounds are formed in the intermediate stages of the oxidation, and these form anhydrides on heating, with condensation and progressive polymerisation. Sulphur activates the oxidation and, in the absence of oxygen, probably dehydrogenates the hydrocarbons, forming highly condensed compounds containing sulphur.

In the cracking process, where oxygen is absent and sulphur may be present only in small amounts, polymerisation and condensation with dehydrogenation and intramolecular change probably produce the asphalt. By this process asphalt may be formed from a paraffin or mixed base crude. A table gives the characteristics of asphalts obtained by "flashing" Kentucky and Mid-Continent residues. A second table compares asphalts from a residue from flashing, one submitted to steam distillation and the other to air blowing at 232° C. The latter has a higher M.Pt. and lower penetration and ductility. A final table compares residues from flashing a Pennsylvania fuel oil, one after blowing with air and the other after blowing with steam.

C. C.

915. Cracking Naphtha at High Pressure. E. B. McConnell. *Oil and Gas J.*, 1931, **29** (50), 16.

The anti-knock value and volatility of gasolines prepared from Mid-Continent, Pennsylvanian and Michigan crudes need improvement for present day market requirements. Cracking operations on gas oil and fuels, which produce gasoline of high anti-knock value, only yield about 50% of the total gasoline. In order to improve the quality of the str. run gasoline, heavy crude naphtha from Mid-Continent crude has been subjected to cracking in a tube and tank unit at a pressure of 1000 lb. sq. in. A pressure of 100 lb. was maintained on separators, towers and accumulators, and the percentage of fresh feed was high enough to prevent vaporization of the light fractions owing to high accumulator temperatures. Operating temperatures were: Accumulator 370° F., coil inlet 575° F., coil outlet 925° F., top reaction chamber 880° F. The heavy naphtha, which represented 15% of the crude, distilled 5% to 212° F. and E.P. 460° F. The yields obtained were 82% distillate, 2% absorption gasoline, 5% tar and 11% gas. The distillate had a high anti-knock value (Octane number 70) distilled 22% to 212° F. with an E.P. of 400° F., and needed only washing with lye to produce finished gasoline. Assuming the heavy naphtha could be marketed after blending with Casinghead (31·8%), the mixture would have an octane number of only 55, 27% distilling to 212° F. and E.P. of 432° F. Blending the distillate with 13·8% of Casinghead gives a fuel of equal volatility at 212° F., but much superior in anti-knock value (octane number 71), E.P. (400° F.), and in percentage distilling over at 284° F.

The method of operation thus offers a solution to the problem of improving the anti-knock value of crude heavy naphtha which may be otherwise unsaleable. R. A. E.

916. Cracking Tests Give Surprising Results. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (48), 30.

There are large stocks of kerosine, gas oil and fuel oil available for use as cracking stock in the States of Oklahoma, Kansas and Missouri.

The results of some recent continuous cracking tests on Mid-Continent kerosine, gas oil and fuel oil are given in order to demonstrate the possibilities. The properties of the oils and the temperatures and pressures employed were:—

	Fuel Oil.	Gas Oil.	Kerosine.
Gravity A.P.I.	24·3°	33·3°	39·3°
I.B.P. °F.	594	448	424
F.B.P. °F.	752	731	590
Viscosity Furol	33 secs. at 122°F.	10 secs. at 77°F.	—
Temp. of cracking °F. ..	922/926	955/967	950
Pressure of cracking in lbs.	250	348	354/355

The yields of products obtained and the gasoline properties were:—

	Fuel Oil.	Gas Oil.	Kerosine.
Gasoline, %	60·1/58·5	64/68·5	74·9/72·7
Flashed Residue, % ..	35·7/37·6	19·2/15·1	6·5/10
Gas (cu. ft./brl.)	442/450	634/698	701/727
F.B.P. of gasoline ..	384° F.	382° F.	386° F.
Octane No. of do. ..	75·5	80	82

The Octane numbers were determined in a "Series 30" Ethyl knock testing apparatus, and show the cracked gasolines to be higher in knock rating than Ethyl Standard motor fuel.

The flashed residues possessed B. S. & W. contents 0·3/0·8%, cold tests 27° F. or lower. R. A. E.

917. "Unrefinable" Oils as Cracking Stock. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (49), 30.

The bulk of the crude oil produced in California is heavy and contains a relatively small percentage of light fractions. Analyses of typical heavy crude oils and fuel oils are given. Although California produced about 25% of the U.S.A. output of crude in 1930, the production of cracked gasoline was only 8·9% of the total. The crude and fuel oils which were analysed have been subjected to cracking at pressures ranging from 100/150 lb. per sq. in and temperatures of 800/860° F. The crudes yielded 44·5/52·1% and the fuels 47·1/61·2% of gasoline, having an E.P. of 437° F. and a high anti-knock value. Yields of 517/1,073 cu. ft. of gas and 38/92 lb. of coke per brl. of charging stock were also obtained. The pressure distillate bottoms distilled about 80% to 572° F., and varied in quantity from 7·3/21·3% of the intake.

By cracking Californian gas oil at a pressure of 275 lb. per sq. in., using the flashed residue method 60% of pressure distillate, 32% of flashed residue and 8% of gas, coke and loss were obtained. After chemical treatment and distillation of the pressure distillate, the final yield of 430° E.P. gasoline was 56%. This gasoline possessed an anti-knock value equivalent to 69 Octane number.

Californian kerosine distillate on cracking to coke at a pressure of 275 lb. per sq. in. and a temperature of 825° F. yielded 86·4% of pressure distillate, together with 650 cu. ft. of gas and 12·9 lb. of coke per brl. of charging stock. After chemical treatment and distillation of the pressure distillate, 64·8% of gasoline and 13·9% of light Diesel oil calculated on the original stock were obtained. The gasoline was water white and non-corrosive, and possessed an E.P. of 437° F. and a high anti-knock value. The composition of this gasoline was determined.

Tables giving analyses of the gasolines and of the pressure distillate bottoms obtained from the various cracking stocks are provided. R. A. E.

918. Process of Cracking Hydrocarbons. I. C. I., Ltd., and W. R. Madel. E.P. 345,664, 24·3·31. Appl., 24.12.29.

The process consists in spraying the oils into an externally heated cracking zone, at a pressure not substantially exceeding the atmospheric, by means of a jet of cracked gases from the process itself, the cracking chamber being maintained at between 650° and 750° C. Cracking takes place in the vapour phase, and claims are also made for processes in which the material is subjected to a separate initial-cracking in the liquid phase. The cracking chamber may be packed with materials such as copper turnings, which exert a catalytic influence on the process. Examples are given which show that cracked gases are superior to nitrogen as a spraying medium.

R. W. L. C.

919. Cracking Petroleum Oils. D. G. Brandt, Assr. to Doherty Research Co. U.S.P. 1,791,114, 3.2.31. Appl., 3.5.27.

A process and plant are described for cracking heavy charging stock by means of hot vapours and highly heated reflux condensate without subjecting it directly to the high temperature of the heating furnace.

The charging stock is brought almost to cracking temperature in coils in a dephlegmating tower, and then passes to a vertical vapour-liquid separator, where it comes in contact with a highly treated reflux condensate which has been passed through a pipe still. The vapours from the separator pass through the main body of oil in a cracking chamber which is fed from the

separator. The oil from the cracking chamber then passes to a concentrator maintained at lower pressure. The heavy residue from the concentrator is used to preheat a supply of non-condensible gas, which is introduced at various points in the system to promote vaporization, and the heavy condensate from the vapours from the concentrator is passed through a separate pipe-still and fed back to the separator. H. B. T.

920. Treating Hydrocarbon Oils. E. E. Stewart. U.S.P. 1,798,338, 31.3.31. Appl., 6.11.26.

The plant described consists of topping and cracking units which may be run in conjunction with or independently of one another. Charging stock is fed through a heater into a vaporizer, in which it meets the vapours from the cracking unit. Any water present in the oil is removed and the lighter vapours pass to a condensing system. Cracking takes place in two stages: in one tower it is liquid phase under high pressure, and in a second tower pressure is reduced and the cracking is in vapour phase. A fractionating column of bubble cap type is incorporated in the plant.
H. B. T.

921. Hydrocarbon-Oil Conversion. C. P. Dubbs and Universal Oil Prod. Co. U.S.P. 1,799,218, 7·4·31. Appl., 30·7·27.

A process for cracking hydrocarbon oil is described. The fresh, untreated oil is introduced to one end of a series of "externally unheated" zones; that is, these zones are not subjected to such heating conditions that the oil passing through would be raised to a conversion temperature. As the oil passes successively through these zones it is maintained at a progressively higher pressure. The unvaporized oil from the last of the series of zones is subjected to cracking conditions of temperature and pressure. The heavier constituents of the cracked oil are removed while the remaining heated conversion products are returned to the zone of the series from which the unvaporized oil was taken off. This causes the vapours released from the oil to move through the series of zones counter-current to the movement of the untreated oil flowing through. A physical co-mingling of the vapours and oils are effected in each of the zones. Vapours which have escaped condensation by the time the initial zone, that is the zone to which fresh oil is added, are subjected to a final condensation and collected. The process results in an equilibrium between vapours and liquids in each zone, whereby the heavier or insufficiently converted fractions of the vapours are condensed and subjected to further treatment.

By maintaining a gradually decreasing pressure in the direction of travel of the vapours, the vapours have an uninterrupted flow after the reduction in pressure, thus eliminating the use of vapour pumps for the purpose of facilitating the passage of the vapours through the oil pools. The most desirable pressure and temperature deferentials between the zones will depend on the nature of the oil and the products desired. W. D. S.

922. Hydrocarbon Oil Treatment. J. B. Heid, Assr. to Universal Oil Prod. Co. U.S.P. 1,799,231, 7.4.31. Appl., 25.4.27.

Hydrocarbon oil is heated to a temperature where substantial vaporization will take place and passed into the upper portion of a vapour separating chamber. The unvaporized portions of the oil are caused to have a successively obstructed descent therein whereby the oil is broken up and vapours are evolved. The residual oil is collected in the lower portion of the chamber. The descent of the released vapours is accelerated by causing them to ascend through passages of successively increased cross-sectional area, the vapours being

removed by means of a pipe situated at the top of the chamber. Spaced decks are provided within the shell, the outer edge of alternate decks being placed flush with the interior wall while the remaining decks are spaced away from the wall. Upwardly projecting tubular extensions are connected to the inner edges of the decks, their cross-sectional area gradually increasing in an upward direction. W. H. T.

923. Lining for Metal Parts of Cracking Plant. L. C. Huff, Assr. to Universal Oil Prod. Co. U.S.P. 1,799,234, 7.4.31. Appl., 21.4.26. Renewed, 7.4.30.

Previous attempts to line the various parts of a cracking plant most subject to corrosion have not been successful owing to the lining buckling and bulging, due to differences in expansion of the lining and the plant wall. The present patent describes a non-corrodible sheet-metal lining provided longitudinally and transversely with spaced expansion folds or flexible joints.

Each section of the lining is achored at its centre to the metal wall of the plant, thus allowing for expansion in all directions up to the expansion folds which bound the section. H. B. T.

924. Producing Lower-Boiling-Point Hydrocarbons. C. P. Dubbs, Assr. to Universal Oil Prod. Co. U.S.P. 1,799,413, 7.4.31. Appl., 11.3.18.

Oil is circulated through a coil and then to and from a group of inclined larger tubes. The heating takes place in the coil and vapours are liberated during their passage through the tubes, ultimately passing to a condenser. Means are provided for regulating the rate of circulation of oil and also the pressure. Advantage may be taken by varying the speed of circulation for settling the free carbon which can be collected and continuously removed. The unvaporized portions of the oil are returned for further treatment into the circulating system. The heating tubes and distilling tubes are positioned in the same furnace, but the latter are so controlled as to be maintained at a lower temperature than that of the former. W. H. T.

925. Cracking Oil. R. T. Pollock, Assr. to Universal Oil Products Co. U.S.P. 1,799,530, 7.4.31. Appl., 24.11.20, renewed 24.3.27.

The plant described consists of a pipe still, expansion chamber, dephlegmator and condenser. The charging stock is pre-heated by passing through the condenser, and is then mixed through an adjustable orifice valve with a definite proportion of reflux from the dephlegmating column before pumping to the pipe still, the temperature conditions in the column being accurately controlled by a water circulating system.

From the pipe still the oil passes to the expansion chamber from which heavy oil containing the carbon formed in cracking is continually drawn off. The oil is subjected to a differential pressure in various parts of the plant which is controlled by valves and gauges. H. B. T.

926. Catalysing Gaseous Reactions. E. B. Miller, Assr. to Silica Gel Corpn. U.S.P. 1,799,858, 7.4.31. Appl., 21.2.25.

Various forms of plant are described embodying the general principle of passing the gas stream, together with a finely divided catalyst, such as silica gel impregnated with a suitable metallic compound, through a converter. The converter is provided with a number of cooling passages fitted with dampers, and the gas stream, before mixing with the catalyst, can be passed through the cooling tubes to remove excess heat generated and thus give a fine control of the temperature of reaction.

From the converter the gases and catalyst pass to a separator, which may be of the cyclone type, and the recovered catalyst is passed through a suitable regenerating system before being reintroduced into the gas stream.

Details are given for the oxidation of SO_2 to SO_3 by means of platinum or copper-iron silica gel, for the hydrogenation of oils by a nickel silica gel and for the cracking of hydrocarbon oils by an iron oxide silica gel.

H. B. T.

927. Process and Apparatus for Treating Petroleum Hydrocarbons. W. M. Cross, assr. to Gasoline Prod. Co. U.S.P. 1,800,436, 14.4.31. Appl., 27.2.28.

The invention relates to improvements in a process and in apparatus for cracking oil under a pressure sufficient to prevent substantial distillation, and to the subsequent recovery of the light fractions. The oil is heated to cracking temperature in a tubular heater and passed to a reaction chamber wherein cracking is consummated. The oil is then conducted to an evaporating stage, which comprises an evaporating and a fractionating tower in which the lighter portion of the cracked product is vaporised and separated. The unvaporized portion is then passed to a separate distilling stage, where additional overhead fractions are removed. The latter distillate fractions are returned to the heating stage for retreatment. The residual oil from the still is passed in a closed cycle to the burners of the heating stage and back to the still. Cycled stock not required by the burners is withdrawn continuously from the system for fuel.

R. W. L. C.

928. Cracking and Distilling Hydrocarbons. J. H. Anderson. U.S.P. 1,800,672, 14.4.31. Appl., 8.10.28.

Apparatus for cracking and distilling solid and liquid hydrocarbons comprises a heating chamber in which a conical retort is rotatably supported with its axis inclined downwards from its larger end. The material to be treated is introduced into the larger end of the retort. The retort is smooth internally and contains a loose mass of metal bodies, which may be of spherical, egg-shaped, cylindrical or irregular form, placed therein to provide agitation and to aid heat transfer during rotation. The products of the operation are discharged through an outlet at the smaller end, where a screen is provided so that the loose metal bodies are retained within the retort.

R. W. L. C.

See also Abstracts Nos. 891, 893.

Hydrogenation.

929. Conversion of Liquid Carbonaceous Materials by Destructive Hydrogenation. N. V. de Bataafsche Petr. Mij. E.P. 345,282, 16.3.31. Appl. 15.10.29.

When treating heavy materials such as petroleum residues or tars at elevated temperatures and pressure with hydrogen in the presence of a catalyst, it frequently happens that the reaction products contain a considerable amount of unconverted products, which may be ascribed to the presence of high boiling or semi-solid or solid substances in the original material. These substances may be of an asphaltic nature and appear to have an unfavourable effect on the reaction.

According to the present invention the process consists in distilling the raw material, if desired in the presence of hydrogen and under pressure to remove the residue containing the deleterious substances and in conducting the distillate without condensation, under pressure and in the presence of hydrogen, over a contact mass prepared from colloidal molybdenum oxide.

The catalyst is prepared according to specification No. 331,199 wherein a suitable absorption carbon is used to support colloidal molybdenum oxide.

R. P.

930. Purification of Mineral Oils, Tars and the Like. I. G. Farbenind. A.-G. E.P. 345,738, 2.4.31. Appl. 23.1.30.

The refining of crude hydrocarbons by the action of concentrated sulphuric acid has the objection that a great loss of the products takes place. To meet the objection it has been proposed to purify by treatment with hydrogen at a high temperature in the presence of a catalyst but certain substances, which tend to resinify or decompose under reaction conditions, reduce the activity of the catalyst. The amounts of these substances are much smaller than the amount of the unsaturated or other constituents which are also removed by concentrated sulphuric acid thereby increasing the refining loss.

By carrying out the purification by stages in such a manner that the substances which decompose when heated up to about 400° C. are first removed without removal of other constituents. The material which has been thus preheated may then be hydrogenated. In many cases even a mild pretreatment is sufficient to avoid difficulties in the catalytic hydrogen treatment.

In the first stage 40% sulphuric acid at a temperature not exceeding 100° C. may be used or alternately small amounts of acetic, oxalic, or malonic acid. Catalysts such as sulphur, potassium bisulphite, zinc chloride, or aluminium chloride may also be used in the pretreatment, or bleaching earth, bauxite or silica gel at higher temperatures from 250 to 400° C. may be used in the liquid or the vapour phase with or without pressure.

In the vapour phase hydrogen, water vapour, air, or other gases may be added.

When employing high boiling raw materials it is preferable to dilute with middle oils.

The duration of the treatment depends on the raw materials under treatment and on the nature of the impurities to be removed. R. P.

931. Manufacture of Liquid Fuels. Cie Internationale pour la Fabrication des Essences et Petroles. E.P. 345,864, 2.4.31. Cons. 15.5.29.

The process is designed for the production of liquid fuels at atmospheric pressure by low temperature distillation or partial gasification by incomplete combustion of carbonaceous materials, and comprises a purification and hydrogenation in the presence of catalysts in a second heat treatment of the vapours after condensation of the condensable products of the first treatment.

R. P.

Chemistry of Petroleum.

932. Induction of the Reaction between Chlorine and Benzene by Ethylene. T. D. Stewart and M. H. Hanson. *J.A.C.S.*, 1931, **53**, 1121.

In the chain mechanism suggested for the reactions involved between ethylene and chlorine an intermediate compound, presumably an activated form of ethylene dichloride has been included. Attempts were made to

get this intermediate to induce a reaction in other systems or itself undergo a reaction with substances other than chlorine. With ethylene chloride and benzene no evidence was found of a Friedelcraft's type of reaction, and from a mixture of benzene, benzoyl chloride, chlorine and ethylene no benzophenone was obtained. However, when benzene was used as a solvent for the chloride-ethylene reaction a mixture of the stereoisomeric forms of benzene hexachloride was formed with the α-form (m. pt. 157) predominating. Hydrogen chloride in considerable amounts was often found among the products and indicated substitution reactions. Equations and mechanisms are given. E. N. H.

933. Decomposition of Hydrocarbons in the Positive Ray Tube. H. R. Stewart and A. R. Olson. *J.A.C.S.*, 1931, **53**, 1236.

Pure hydrocarbons up to and including hexane were prepared, and positive ray analyses were made of each hydrocarbon. Peaks due to ions of carbon chains shorter than the original were found in every case except that of methane.

With hexane it was shown that exposure to a hot filament led to thermal decomposition during analysis. A new apparatus was designed to minimise thermal decomposition and details of this are given. The peaks were still observed and were as well marked as with the original apparatus.

After a consideration and elimination of possible explanations one remained, that the ionising electrons decompose the hydrocarbons on impact. Using propane has an example the equations given are :—

$$C_3H_8 \longrightarrow C_3H_5^+ + E^-$$
$$C_3H_8 \longrightarrow C_2H_5^+ + CH_3 + E^-$$
$$C_3H_8 \longrightarrow C_2H_3^+ + CH_3^+ + E^-$$
$$C_3H_8 \longrightarrow C_3H_7^+ + H + E^-$$

To explain the multiple sub-peaks of propane and butane it seems necessary to assume that a single electron can eject a number of hydrogen atoms. E. N. H.

934. Physical Properties of the Normal Paraffin Hydrocarbons, Pentane to Dodecane. A. F. Shepard, A. L. Henne and T. Midgley, Jr. *J.A.C.S.*, 1931, **53**, 1948–1958.

The normal paraffins used for the investigation were all prepared from a special gasoline containing approximately 70 per cent. paraffin hydrocarbons, by the method previously described by Shepard and Henne (*Ind. Eng. Chem.*, 1930, **22**, 356). After a rough fractionation, each cut was treated with chlorsulphonic acid until no further change in the density of the hydrocarbon layer could be detected. Final purification was accomplished by careful fractionation through a flat spiral fractionating column equipped with an efficient dephlegmator. The chlorsulphonic acid treatment is a lengthy process, and as long as 20–56 days was required, renewal of the acid being necessary every 3–4 days. The normal paraffins left by this treatment ranged from 23–48% of the hydrocarbon originally taken. The boiling range (distillation curve) and the total freezing range (freezing curve) were the two criteria used to indicate the purity of the materials obtained. Both methods indicated a high degree of purity in the products. It was noticed that undecane and dodecane were not absolutely stable when distilled under atmospheric pressure, indications of cracking being shown by the brown colour obtained in treatment of the distillates with sulphuric acid. Besides

boiling and freezing points, figures were obtained for the densities, refractive indices, molecular refractions and dispersions, the critical solution temperatures (aniline points) and the viscosities. It was found that the empirical relationship $T\frac{\sqrt{MR}}{M-MR}$ = constant, proposed by Nerasov involving the boiling point, in °K, the molecular weight, M, and the molecular refraction, MR, calculated from the density and the refractive index by means of the Lorentz-Lorenz formula gave figures in close agreement with those obtained experimentally. S. F. B.

935. Petroleum and Alpha Radiation. S. C. Lind. *Nature*, 1931, 127, 813–814.

Hydrogen is the most abundant gas produced by the action of α particles on saturated hydrocarbons, but it does not combine further with unsaturates under the same influence. This is important in the possible *rôle* of α radiation in the interaction of hydrocarbons in the earth's crust. Free hydrogen is, however, generally absent from natural gas. If the origin of petroleum is partly radioactive, some theory to account for the absorption of the hydrogen must be advanced. It is suggested that the complete catalytic removal of hydrogen under high pressure and temperature may occur if the hydrogen is produced by the action of α particles on unsaturated hydrocarbons in the first place (since less hydrogen is formed than from saturated compounds).

It is concluded that while the thermal interaction of hydrocarbons at high pressures and temperatures (suggested by Wilson) may be the more prolific source of petroleum, the radioactive origin cannot be excluded. Some radioactive material has been found associated with crude petroleum, and it is certain that hydrocarbons will be modified in the earth's crust when exposed to α rays, the reaction being a condensation to higher members with elimination of hydrogen and methane. C. C.

936. Separation of Normal Octane from Petroleum by Distillation and Crystallation. R. T. Leslie and S. T. Schicktanz. *Bur. Standards J. Research*, 1931, 6, 377–86.

A fraction boiling between 100° and 300° from an Oklahoma crude oil was subjected to an interlocking process of distillation and crystallisation to separate octane. The method and apparatus are described in detail. The b. pt., f. pt., refractive index and mol. wt. of the product are given and compared with the properties of a carefully purified sample of synthetic octane. It is estimated that not less than 1% of octane was present in the oil investigated. W. S. E. C.

Analysis and Testing.

937. Laboratory Distillation Apparatus. H. Burstin and J. Winkler. *Chem.-Ztg.*, 1931, 55, 212.

The apparatus described was designed for the continuous vacuum distillation of oil residues, but is suitable also for the treatment of easily decomposable or foaming liquids, such as phenols and sulphonic acids and for organic liquids containing water. It consists essentially of a heating chamber in which the

liquid is vaporised as it enters through a needle valve, a dephlegmator packed with Raschig rings and a condenser; the heating surface and vapour space are large compared with the quantity of material heated, and the latter is therefore maintained at a high temperature for only a short period.

W. S. E. C.

938. Comparison of Gum Test Methods. J. C. Molitor. *Oil and Gas J.*, 1931, **29** (51), 152.

The data on the gum content of gasolines given by various methods described by Bridgeman and Aldrich (*see Abs.* 589) is confirmed by the Vacuum Oil Co.'s Laboratory, who, however, point out that no information is given on the steam oven method.

Comparative tests by this method and the air jet method showed that the steam oven method gave results about 35% of those given by the air jet method, and was slightly more easily reproducible. Blends of 50% of high gum content gasoline with 50% gum free gasoline gave values on an average of 37% of the original gasoline, as compared with 44% in the case of the air jet method.

Extrapolation of the curve showing gum contents of different volumes of gasoline evaporated, to zero (indicating instantaneous evaporation), gives values of two-thirds of the gum content at 50 c.c. evaporated by the air jet method. The results are considerably higher than those obtained by the steam oven method, indicating that the latter gives results more nearer the true gum content at instantaneous evaporation.

C. L. G.

939. Examination of Asphalt and Tar with the Quartz Analysis Lamp. F. Macht, *Erdöl und Teer*, 1931, **7**, 225–227.

The use of the quartz analysis lamp for the differentiation of asphalts and tars and mixtures of these is discussed. If the material is placed in a thin layer on a glass plate and exposed to ultra-violet light, asphalt gives a greenish-yellow colour and tar a dark brown colour with red cast. The higher the oil contents the brighter are the colours.

If solutions in benzol or benzine are employed the effects are rather different. Mexican asphalt in benzol shows a blue-green colour, while Galician bitumens show a yellow grey. Tars in benzol solution give yellow colours. The method of allowing a drop of the benzol or benzine to fall on a filter paper or clay plate is recommended. The appearance of the spot is bright brown for asphalts and yellow for tars, the colours being darker with benzol solutions. The difference between the colours given by asphalts and tars is very marked and enables admixtures to be determined, with practice, to about 5%, particularly in the range 5 to 30% asphalt. The yellow colour shown by tars is due mainly to anthracene.

E. B. E.

940. Determination of the Toluene Content of a Mid-Continent Petroleum. J. H. Brusen, R. T. Leslie and S. T. Schicktanz. *Bur. Stand. J. Res.*, 1931, **6**, 363–367.

Commercial petroleum fractions from an Oklahoma crude oil were fractionated into 1° cuts and tested for toluene by nitrating to 2·4-dinitrotoluene by means of a nitrating method which was found not to attack the other constituents of the fractions. Toluene was found in all the cuts boiling between 90° and 112°. The maximum concentration (32·2%) was found in the fraction boiling between 107° and 108°. Total toluene present amounted to ½% based on the crude petroleum.

W. S. E. C.

941. Analysis of Petroleum and its Distillates for Reducible Substances and Adsorbable Matter by Means of the Polarographic Method with the Dropping Mercury Cathode. B. Cosman and J. Heyrovsky. *Trans. Electrochem. Soc.*, 1931, **59**, 23 pp.

The electroanalytic method with the dropping mercury cathode and automatic polarographic recording of the current-voltage curves has been applied to the estimation of (1) electrolytically reducible substances, and (2) adsorbable (surface-active) matter in petroleum and its distillates. The analysis of electro-reducible substances is carried out by shaking the petroleum with a solution of ammonium chloride and submitting the aqueous extract to a polarographic examination after the atmospheric oxygen has been expelled by hydrogen or by the addition of a sulphite. About 0·005% of reducible substances may be detected in this way.

Many types of petroleum were found to contain acidic substances, probably naphthenic acids which may be extracted by aqueous alkaline solutions and which possess a prominent anticatalytic action in the reaction between oxygen and sulphite. These anticatalysts suppress maxima occurring on current-voltage curves at positive potentials, thus indicating their negative charge and strong adsorbability. The content of adsorbable substances is determined by adding the petroleum or its solution in methyl alcohol from a microburette to an acidified methyl alcohol solution of copper sulphate. The maximum on the current-voltage curve, due to the latter solution, is hereby depressed and the dilution at which a half-suppression is obtained is taken as characteristic for the adsorbability in the petroleum cut. Naphtha from different sources has been found to contain the least adsorbable substances (dilution number 10–400), then gasoline (400–2000) and lubricating oil (3000–13,000). Crude oil is most adsorbable with dilution numbers up to 41,000. W. S. E. C.

942. Continuously Indicating and Recording Viscometers. J. L. Hodgson and H. E. Dall. E.P. 345,210, 16.3.31. Appl., 14.12.29.

The apparatus described consists of a vessel pivotally suspended from a pendulum weighted beam or other weighing device acting as an indicating or recording mechanism. Liquid, maintained at a constant temperature, is supplied to this vessel at a constant rate of volume flow in a horizontal direction by means of a pump running at a constant speed or an orifice discharging under a constant head. The liquid discharges through a tube at a sufficiently small difference of pressure to enable the flow to be laminar.
W. S. E. C.

See also Abstracts Nos. 852, 855.

Engines.

943. Induction Conditions, Distribution and Turbulence in Petrol Engines. S. J. Davies. *Engineering*, 1931, **131**, 57–60, 89–91.

The paper deals with tests on a 4-cyl. petrol engine to determine the effect of induction conditions, distribution and turbulence on air and fuel efficiencies and general performance of the engine. Special valves were used to modify the turbulence by reducing the effective port area. Some of these valves were so constructed that the direction of the inflowing charge could be varied. With this method of controlling the turbulence the timing and complete valve opening diagram remained constant throughout all tests.

The results show that increased turbulence caused by greater inlet velocity improves the thermal efficiency, and that increased turbulence caused by changes in the direction of the inflowing stream benefits the performance. Tests with heated induction air showed that improvements in distribution follows by no means uniformly from the addition of heat. The reduction in volumetric efficiency and output is in direct relationship with the quantity of heat added, and the "combustion chamber efficiency" is not necessarily constant, but may be affected markedly by changes in the temperature of the induction air. J. M. F.

944. The Present Position of the Diesel Engine for Marine Purposes. H. H. Blache. *Mar. Eng.*, 1931, **54**, 136–138.

Although a uniform design has not been reached, all marine diesel engines are similar in their lower structures, and in that forced lubrication and enclosed crank casings have been adopted. Diesel dynamos generating electrical current for driving auxiliaries are general excepting for tankers.

The 4-str. engine has progressed in the direction of admitting the inlet air under a pressure of from 4 to 5 lb./sq. in. (high pressure induction) as distinct from the low-pressure induction (air at about 0·7 lb./sq. in.).

The Burmeister and Wain type of pressure induction 4-str. diesel engine embodies a special design of inlet valve whereby the cylinders are filled with atmospheric air during the principal part of the stroke in the usual manner. A movement of the multi-port valve and casing provides high pressure induction during the closing period and the opening period of the inlet valve; the compression space being scavenged at the top of the stroke.

The various types of 2-str. diesel engines can be classified according to the manner in which the cylinders are scavenged. The "non-complicated" type has exhaust and scavenging ports controlled by the main piston (not largely used), which permit the disadvantageous pollution of the scavenging by the exhaust gases due to blow-back from the exhaust. In the "complicated" type, a complete and clean scavenging of the cylinders is possible, "blow-back" from the exhaust being prevented.

Mechanical injection of fuel is now the general rule though fuel pumps and valves vary in details of design. The adoption of this in place of air injection has resulted in a reduction of about 6% in the fuel consumption per b.h.p.

In so far as clean air is essential to obtain a smokeless combustion with airless injection, the Diesel engine is fitted with high pressure induction (in the case of four-stroke engines), and the "complicated" two-stroke engines are to be preferred. Moreover, the importance of airless injection for marine purposes is its adaptability for the different qualities of fuel oil obtained all over the world. C. I. K.

ABSTRACTS.

Geology.

945. Alberta Syncline, Canada. T. A. Link. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 491-507.

A consideration of the geological history of the Alberta syncline indicates that the western portion adjoining the Rocky Mountains has been a zone of great sedimentation throughout Palæozoic, Mesozoic and Tertiary times. This geosyncline of deposition appears to have travelled gradually eastwards, but so far as the Mesozoic and later sediments are concerned the area of maximum deposition appears to lie close to the foothills of the Rockies, not far from the Turner Valley field. The stages in the development of the regional structure are discussed and the most recent data on thicknesses of formations are used to support the theory of westerly thickening in the basin.

In the accumulation of gas and oil in the flanks of the Alberta syncline a source of pressure would be found in the hydrostatic head of the Sweet Grass Arch and also the east flanks of the basin. On the west, thrusting probably limits the development of a hydrostatic pressure by breaking the continuity of the formations, but this thrusting may itself be the source of pressure in the Turner Valley field. Various possibilities with regard to the direction of oil migration are discussed and inferences drawn as to their effects on the location of oil and gas pools. V. C. I.

946. Cap Rock Petrography. L. S. Brown. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 509-529.

An account of the study of a large number of well cores of the cap rocks at the Hoskins Mound and Bryan Heights salt domes.

The brecciation, faulting and slickensiding in the cap rock series, and the shear banding in the anhydrite, indicates that the salt structures are due mainly to upthrust under enormous pressure of the salt and cap as a vertical intrusion.

The anhydrite cap is considered to be a true sedimentary formation and the identity in structure, texture and the common included minerals in all the caps studied suggest that they are parts of the same formation, the Comanchian.

The calcite of the calcite cap is considered to be secondary, due to the alteration of the underlying anhydrite by meteoric waters containing carbonates. The gypsum is also thought to be due to the hydration of anhydrite by circulating waters.

Part of the sulphur is primary, but the commercial deposits appear to be due to secondary segregation. V. C. I.

947. Tertiary History of the Sabine Uplift. C. L. Moody. *Bull. Amer. Assoc. Petr. Geo.*, 1931, **15**, 531-551.

This paper traces the history of deposition in the Gulf Embayment in later Mesozoic and early Tertiary times. The dawn of the Tertiary commences with a marked transgression which was followed by alternate retreats and advances of the sea with a gradual tendency to progressive emergence of

the land mass. The Oligocene deposition was much more limited than that of the Eocene, while the Miocene formations are largely continental. The stages in the history of deposition are indicated by a complete series of diagrams.

After the Miocene deposition the north Louisianan area was uplifted differentially and the main structural features were finally achieved. The interior salt domes had originated as early as Mesozoic times, but they were largely upthrust at this later stage and the main fault features of the area are also post Miocene.

The cause of these later movements is considered. Both lateral pressure and isostatic adjustment due to overloading are rejected as possible explanations. It is suggested that the most probable cause lies in the lateral migration of molten magmatic material in the underlying crust. V. C. I.

948. Account of Early Endeavours on Anticlinal Theory in Canada. R. B. Harkness. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 597-610.

This contains a series of references to the early discovery of oil in Canada and indicates that as early as 1840 the Geological Survey had investigated several seepages of oil in Ontario. Full credit is given to the work of Logan, Murray and Hunt, particularly the latter; and it is shown that already in 1861 Hunt had formed a clear idea of the essentials of the anticlinal theory of oil occurrence.

The paper gives long extracts from Hunt's publications which substantiate the claims of this worker to recognition as the originator of the anticlinal theory. V. C. I.

949. Chemical Considerations regarding Origin of Petroleum. B. T. Brooks. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 611-627.

A review of the chemical work on the origin of petroleum. It is claimed that the geological evidence does not support the theory of heat decomposition within the earth's crust and that the chemical evidence on this point when considered in full also fails to support Engler's theory of heat decomposition. The claim that alpha radiation may be an agent in petroleum formation is negatived by the absence of hydrogen in natural gas, a factor which also precludes the possibility of high pressure hydrogenation as a means of producing petroleum in the crust.

Evidence is submitted that many types of sedimentary rocks, in addition to fullers earth, are capable of polymerising the unsaturated hydrocarbons, and this may account for the gas oil and lubricant fractions in crude oil. It is claimed that the formation of hydrocarbons is a low temperature process. Reduction by an aerobic bacteria or hydrogenation under natural conditions coupled with polymerisation are considered to be the most probable cause, the raw material being the diverse organic matter entombed in the sediments. V. C. I.

950. Petroleum Possibilities of Turkey. D. Eyoub. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 629-670.

The petroliferous provinces of Turkey are divided into three groups:—(1) Mardin region; (2) Anatolian Plateau; and (3) Miscellaneous Coastal. With the exception of the region bordering Iraq and Syria, most of the localities in these provinces are considered too remote or unfavourable geologically for the economic production of oil.

The *Mardin region* adjoins Persia, Iraq and Syria, and the following formations are present: Pleistocene and Recent, Pliocene, Miocene, Oligocene, Eocene, Cretaceous and Palæozoic. The beds have a regional

dip to the south, the major structural axes being E.W. Strong overthrusts are present, but most structures are asymmetrical anticlines with their southern flanks much steeper than their northern. Many of the south limbs are vertical and some faulted. Liquid oil seepages, asphalt deposits and H_2S emanations occur in many places. Though there has been no petroleum developments in this region, the conditions are such that it is not unreasonable to consider that certain areas have decided possibilities of producing oil in commercial quantities.

A concise geological account is given of the possible oil-producing localities in the *Anatolian Plateau*, which is a large basin broken by faults. The rocks vary in age from Quaternary to Pre-Cretaceous; igneous rocks are also present. Earth movements (which are of recent date) and igneous activity have been very intense.

The regions occurring in the *Miscellaneous Coastal* group (Murefte, Chirali, Boyabot and Mapavri) are also described in detail. Similar formations are present and faulting is again a very prominent structural feature.

The author states that the present petroleum laws (of 1914), which have hitherto handicapped development, are now being modified.

The paper is well illustrated by topographical and geological maps and sections. G. S. S.

951. Method for determining the Effective Porosity of a Reservoir-Rock. K. B. Barnes. *Penns. State Coll. Bull.* (*Min. Ind. Exper. Stn. Bull.* 10), 1931, 1–12.

This is a method recently developed which, from a petroleum-engineering point of view, is claimed to have advantages over other procedures.

The effective porosity of the reservoir rock can be determined instead of the absolute porosity. Time is also saved in the actual testing of the samples. Error due to loss in grinding material to grain size (which has been estimated to average about 0.08%) is eliminated. Moreover, samples after testing are in the same condition and shape as before and can be kept for further examination or for filing.

A complete calculation for porosity and tables of other data are also given in the paper. G. S. S.

952. Gas Field of Daszawa, Poland. O. C. Wyszinski. *Rev. Petrolifere*, 1931 (429), 785.

The Daszawa gasfield is situated about 40 metres east of Boryslaw in Poland and lies in front of the highly folded Carpathians, between the latter and the Cretaceous plateau of Podolia.

The geology is masked largely by alluvium, but the general structure appears to be a gentle anticlinal fold composed of marls, clays and fine sands of Tortonien age. This anticline extends in a N.N.E.-S.S.W. direction; there are six horizons at which gas is found, but the principal one lies at a depth of from 740 to 780 metres and gives gas wells with a capacity of from 200 to 300 cu.m. per minute.

The production of gas in this small field since 1923 has exceeded 400,000,000 cu.m., and the present production is about 95,000,000 cu.m. per year. V. C. I.

953. East Texas Oil along Old Shore Line. E. G. Woodruff. *Oil and Gas J.*, 1931, **30** (6), 15.

This field embraces the western part of Gregg Co., N.W. Rusk Co., and narrow portions of S. Upshur and S.E. Smith Counties. It has a maximum width of over seven miles and a minimum of over three miles.

The geological formations are Cretaceous and Tertiary in age and include the Georgetown Limestone, Woodbine Sands, Austin Chalk and Pecan Gap Series.

The accumulation of the oil is along an old shore line. Elevation in Woodbine times of a large circular island resulted in the washing down and redepositing of the sandstones along the shore, and these now form the upper sands which are oil bearing. This movement and subsequent erosion caused the Woodbine to be slightly tilted against the Georgetown high sand, and above these occur irregular beds of shale and sand also dipping westward. Both below and overlying these strata are impervious rocks which thus prevent the escape of the oil.

Several thousand oil and gas wells have been completed over the uplift in the Shreveport region, and the main part of this structure has been a prolific producer. There are 100,000 productive acres, and probably the area is one great pool with not a dry location in the entire field.

Owing to the varying thickness and extent of the sands, the total recovery cannot be estimated with any degree of certainty; some put the recovery at 7,500 brls. per acre, and others as high as 25,000; the author gives 15,000 brls. as a conservative estimate, in which case the area will produce 1,500,000,000 brls. Thus this field alone can supply the United States for two years. G. S. S.

Field Technology.

954. Using Pressure Drop in Sand to Measure Well Capacities. H. D. Wilde, Jnr. and T. V. Moore. *Oil Weekly*, 1931, **61** (12) 26.

The production of oil at a rate less than the maximum, of which the pool is capable, may be necessary for a number of reasons, such as curtailment to meet market demand, to conform to a proration programme, or voluntarily to decrease operation costs and increase ultimate production. The curtailment should, however, be equitable to all the individuals owning various parts of the pool, should not damage the pool, should not interfere with economical operations and the allowances under it should be susceptible of rapid and accurate determination.

The simplest method in use at present is on a time basis under which each well is allotted a given period to produce at its maximum rate and is then shut in for another given period. This plan, although reasonably equitable, is affected by several disadvantages. Where wells are allowed to produce under wide open flows, the ultimate production is less than where the wells are produced at lower rates, and this is particularly true where water can move in toward the wells. As a consequence, production at the maximum rate is harmful to the pool. Investments in the wells are unnecessarily increased due to the fact that the large capacity lines, separators, etc., are used for a fraction of the time only. Operators are encouraged to drill more wells, since the more wells there are on a given tract the greater production the tract will be allowed, and this results in excessive outlay.

Another method is on an average basis under which operators are permitted to produce at a steady rate a fraction of the total production equal to the fraction that their productive acreage bears to the total productive acreage.

This results in an inequitable distribution, as the oil sand will invariably show a variation in thickness, porosity, permeability and saturation in different parts of the pool.

The "Potential Production" basis method of prorating also shows serious disadvantages. Under this method each operator is permitted to produce in the ratio that an open flow test of his well bears to the total open flow production. The open flow test is a measure of the capacity of the property to produce in its existing condition but it is not proportional to the amount of recoverable oil under a property. In addition the open flow test is materially affected by the size of the tubing or casing used in the well, the size and length of the surface flow lines, the depth of the well and the gas-oil ratio.

A method widely used at the moment is based on an average and maximum capacity basis. The field is divided up into a number of units of equal area and the average potential production of the unit is determined by summing up the potential production of all wells on the unit and dividing by the number of wells. This is reasonably equitable but the disadvantage of the open flow test of the wells still remains.

The methods described fail to satisfy fully the four requirements mentioned earlier although the last method satisfies them reasonably well.

The true measure of the rate at which wells are capable of producing must depend upon the character of the producing formation and the conditions under which the fluids are found. The allowable production should not be influenced by the equipment which happens to be installed in the well.

The method suggested by the authors to meet this need is as follows :—

The field is divided into units of equal area and 25% of the total production distributed equally among the various units. The remaining 75% is divided among the units in proportion to their capacity to produce oil. This capacity is determined from curves showing the relation between bottom hole pressure and production rate. Since the pressure differential in the sand is the force which causes the flow of oil to the well, an equitable method of distribution would allow all operators to produce oil under a driving force which is a certain percentage of the total driving force available. The allowance for a given well is then the rate at which that well can produce oil when the pressure drop in the formation is a definite percentage of the rock pressure and this percentage is fixed for the whole field.

The method will depend upon the rapid and accurate measurement of bottom hole pressure.

The advantages of this proposed system are :—1. Pressure drop in the sand furnishes a measure of the capacity of the well superior to the open flow test ; 2. Equipment has no bearing upon the allowable production ; 3. Reduction of the tendency to withdraw oil from a neighbouring territory by establishment of substantially uniform pressure drops in the sand ; 4. Control of pressure drops reduces tendency to form low pressure areas and gas channels ; 5. Data required can be taken at normal rates of flow.

The disadvantages are :—1. Increase in labour and time in securing necessary data ; 2. Experimental error involved in measuring bottom hole pressure.

L. V. W. C.

955. Well Method of Producing Oil. W. P. Haseman. *Petr. Eng.* 1931, **2** (9), 152.

The three parts of an oil well, the well bore hole, the porous rock reservoir, and the compressed fluids contained therein, form a working and producing mechanism for the transference and transformation of the energy stored in the compressed fluids to the production of oil. The efficiency of this transference and transformation depends upon two factors, the spacing of wells and the conservation and utilization of the energy and gas in the reservoir.

The well spacing formula advanced by the author is based upon a conception which classifies a producing sand in accordance with the ability of the stored energy to produce the oil from the sand with a set standard distance between the wells.

A sand has a producing quality defined as "1," when say 5% of all the oil confined in the rock reservoir is produced with a spacing of one well to each 10-acre tract. It has a producing quality "2" when 10% of the oil is produced with a spacing of one well to each 10-acre tract. Similarly for quality "3" when 15% is produced, "4" when 20% and so on.

Zero wells on a 10-acre tract will produce zero oil, while an infinite number of wells to each 10-acre tract will not produce more than 100% of the oil. Available data indicates that an infinite number of wells on a 10-acre tract will not produce much in excess of 50% of the oil confined in the reservoir. Thus in the case of a sand of quality "2" zero wells to each 10 acres will produce zero oil, one well to each 10 acres will produce 10% of the oil and an infinite number of wells to each 10 acres will produce 50% of the oil. A graph drawn through these three points will indicate the per cent. of oil produced from the given sand at every intermediate point.

The method of operation of the wells will influence the degree of conservation and utilization of the gas energy with a given spacing of the wells.

The establishment and maintenance of flow of oil and gas to and out of the well is due to a release of energy from the reservoir, and the rate of release of this energy is considered as directly proportional to rates of release of oil and gas from the reservoir. The efficiency of the well mechanism in producing oil under these assumed conditions is taken as directly proportional to the height of the lift and inversely proportional to the gas oil ratio. Also this efficiency is further defined as 100% when the energy spent by the well reservoir in lifting a barrel of oil vertically against gravity to the height of the lift is equal to the energy released by the volume of gas which accompanies the barrel of oil and expands isothermally from a pressure of 0.34H to 15 lbs. per sq. in. where H is the height of the lift.

Three examples are given showing the efficiencies and gas volumes utilized. From the data obtained it is assumed that with correct spacing of wells and correct rate and methods of production, the development and production of oil and gas can be controlled scientifically and efficiently. L. V. W. C.

956. Behaviour of Mud Pumps. H. E. Hayes. *Oil Weekly*, 1931, **61** (12), 32.

The majority of rotary rigs use steam as the power for driving the pumps, and in general 2 double-acting 2-cyl. slush pumps are installed. These pumps are of the duplex type designed for maximum working pressure of 1000, 2000 or 3000 lbs./sq. in. and operate on boiler pressures ranging from 125 to 225 lbs./sq. in. Where the motive power is other than steam the steam cylinders are replaced by a power end and the pumps are power driven.

Circulation is to be maintained at a reasonable rate during drilling, and the mud flow relation will be given by $Q = KP^n$, where Q is the flow in cu. ft./min., P the pressure in lbs./sq. in. and K is a constant. The exponential "n" will vary slightly from day to day, depending on the well conditions, but will range from 0.500 to 0.525.

Assuming an exponential of 0.5 the expression given earlier becomes $Q = KP^{0.5}$ and it will be seen that the pressure required varies as the square of the change in flow, and intensity as the square of the change in the constant K. The hydraulic horse power varies as the cube of the change in flow and inversely as the square of the change in the constant K.

Neglecting the initial or shearing pressure, each drilling condition will have a particular value of K. This value will vary from 0, in pressure tests, to 9 in low resistance cement jobs.

Given the value of K, flow curves may be plotted for various stated drilling conditions.

The mud rate that will occur against a given drilling condition depends upon the behaviour of pressure characteristic of the pump. The form of this characteristic depends upon the speed torque characteristic of the prime mover, and the magnitude depends upon the capacity installed, the speed ratio and the size of the liners. By plotting the pump characteristic over the flow condition curves it is possible to ascertain the operating pressure and volume under a given condition.

Curves drawn for the steam pumps show relatively high volumes of mud at low pressures and low mud volumes at high pressure. Crossing of the curves of two pump operation indicates that at the lower pressures the parallel connection will give the greater flow. At the higher pressures the greater flow will be obtained with the series compounded connection. Curves of the alternating current motored pumps indicate medium mud rate at lower pressures and a slight decrease in mud rate at higher pressures.

Parallel operation of mud pumps is employed on low resistance cementing operations and at times when the mud flow with one pump is deemed insufficient for the purpose in hand.

The capacity of the pump, expressed in hydraulic horse power, depends upon the pressure-volume characteristic and is, therefore, dependent on the behaviour.

A study of the relation of pressure volume characteristics of mud pumps to drilling operation and flow conditions may lead to a better adaptation of the various types of prime movers to secure economy of operation and the best combination of suitability. L. V. W. C.

957. Origin and Removal of Pipe Line Dust. D. A. Sillers. *Oil & Gas J.*, 1931, **29** (52), 63.

Pipeline dust originates from :—(*a*) fine well sand ; (*b*) adventitious matter picked up during construction ; (*c*) mill scale, rust and products of corrosion.

Dust is deposited when changing over oil lines to natural gas, which dries out the oil from the oil and dirt lining left on the pipe. Excess of dust causes the cutting out of compressor valves and reducing valves, meters and fittings, and clogs up the orifice meters. It can be eliminated by :—(1) reducing the velocity of gas, allowing the dirt to settle out, at the same time increasing the diameter of the pipe, and keeping up the rate of flow ; (2) cleaning by centrifugal force ; (3) filtering through cotton bag strainers, or through layers of oiled excelsior, hair, sea moss, etc. ; (4) oil contact cleaners, where a spray of oil meets the gas and absorbs the dust, the gas being separated in an upper chamber in the cleaner. The efficiency of this type of apparatus is practically 100%. C. L. G.

958. Pipe Line Symposium. *Oil & Gas J.*, 1931, 30 (3).

A 162-page symposium on pipe lines, giving the trend of pipe line construction in various parts of the States, pumping methods and problems, and many maps of the location of the various pipe line sytems. A large map of the United States is included showing the Natural-Gas Trunk Line systems and giving statistics of the production of natural gas, length of gas pipe lines, etc. The Bureau of Standards tests for determining the corrosivity of soils are described and articles on the protection of lines from corrosion given. Reconditioning and welding of lines is dealt with and methods for odorising natural gas discussed. C. L. G.

Crude Oil.

959. High Gasoline Yield from Pettus Crude. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **29** (48), 30.

Crude from the Pettus Field, South-west Texas, yields 47% of straight-run gasoline having an I.B.P. of 122° F., 20% over at 216° F., F.B.P. 399° F., sulphur content 0·01%, octane number (S.30 engine) 46. The topped residue (52·2%) was subjected to cracking, using the "flashed residue" method of operation, at a pressure of 348 lb., and at a temperature of 940° F. By this means 73% of cracked gasoline having an I.B.P. of 83° F., 20% over at 178° F., F.B.P. 392° F. and octane number of 73, was obtained. Blending the two gasolines in the proportions in which they were produced from the crude oil gave a total yield of 85·1% of gasoline having the following properties :—Grav. A.P.I. 58·3°, I.B.P. 101° F., 20% over at 199° F., F.B.P. 395° F., Octane number 65.

The topped residue was very stable to distillation at atmospheric pressure, no material decomposition taking place at a temperature of 760° F., when 95% distilled over. In addition to the cracked gasoline, 13·8% of flashed residue (visc. Furol at 122° F.=53 sec.), and 513 c. ft. of gas per brl. of charging stock were obtained from the topped residue. R. A. E.

960. Topping and Cracking Van Texas Crude. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **30** (5), 22.

Crude oil from the Van Pool of Van Zandt County, East Texas, is produced from a depth of 3,400/3,600 ft., and is a paraffin base oil, having an A.P.I. grav. of 34.6°, sulphur content 0·9%, viscosity Furol at 77° F. 40 sec., Cold Test 0° F. The results obtained from an atmospheric and vacuum distillation of the crude on the laboratory scale are shown in tabular form. By topping the crude a yield of 27·5% of straight-run gasoline having the following properties was obtained :—A.P.I. grav. 61·4°, I.B.P. 96° F., F.B.P. 400° F., sulphur content, 0·07%, anti-knock value in S.30 engine equivalent to an octane number of 43, paraffin hydrocarbon content 95·4%. The gasoline thus has a higher paraffin content and a lower anti-knock value than Pennsylvanian straight-run gasoline of similar F.B.P. The topped residue (71.5% yield ex crude) possessed an A.P.I. grav. of 24.3°, viscosity Furol at 77° F. 52 sec., and was subjected to cracking at transfer temperatures of 910/935° F. under a pressure of 350 lb. The process was regulated according to the quality of the residue produced, nine separate tests being carried out. This residue in certain tests respectively satisfied the requirements of Bunker A, B and C fuel oil specifications, and in the last test the residue possessed a viscosity Furol of 2,780 sec. at 122° F. The yield of cracked gasoline varied from 36/62% of the cracking stock (equivalent to 25·7/44·2% of the original crude oil) according to the quality of the residue. The properties of the gasolines varied within the following limits :—A.P.I. grav. 58·3°/60·9°, I.B.P. 84/97° F., 20% over at 179/188° F., F.B.P. 394/410° F., Octane number 70/73. The properties of the cracked residues are tabulated and graphs provided to show the relation between the viscosities and gravities of the residues and the yields of cracked gasoline. The cracked residue, having a viscosity of 2,780 sec. Furol at 122° F., was distilled to coke, of which 25·8/26% by weight was obtained, and analyses of the overhead distillates are provided. The quantities of incondensable gas produced during cracking ranged from 210/378 cu. ft. per brl. of charging stock and the gas contained approx. 2 gal. of gasoline per 1,000 cu. ft. R. A. E.

961. Refining Value of East Texas Crude. W. T. Ziegenhain. *Oil and Gas J.*, 1931, **29** (49), 24.

East Texas Crude is of relatively high refining value, being quite equal to average Mid-Continent crude, although producing a fuel oil of high cold test. This is not of much importance to large refiners with cracking equipment, and is balanced by the yield of gasoline of high anti-knock value.

Plant yields on the crude were as follows: Gasoline (415 E.P.) 37% Kerosine (41-43 gravity) 6%; Gas oil (29-30 gravity) 24%; Fuel (19 gravity, 110 cold test) 30%. This corresponds very closely with the yields from Seminole crude, but owing to its lower initial cost in the field—25 cents. per brl. as compared with 65 cents. for Seminole—it is more profitable to work up.

The results of laboratory distillations on East Texas crudes from (*a*) Longview-Joiner area, and (*b*) Henderson area are given. The lub. oil distillates from these crudes, although having pour points of 75 and 80° F. appear to be quite suitable for working up further into the various grades.

Details are given of the cost of the oil in the fields and the various transportation systems through which the oil can be supplied. C. L. G.

962. Joiner Yields 76% Gasoline. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **30** (1), 40.

Crude from Joiner field, Rusk County, East Texas is obtained at an average depth of 3,600 ft.; the producing sand is about 25 ft. thick with chalk above and limestone below. The sample of crude examined had an A.P.I. grav. of 28·9°, 0·23% sulphur, 54% over at 572°F. By atmospheric distillation gasolines of E.P. 384°, 401° and 437°F. were obtained and examined in an engine. The knock ratings determined were equivalent to octane numbers of 62, 58 and 50 respectively. The straight-run gasoline contains 1·8% aromatic and unsaturated, 31·4% naphthenic and 66·8% of paraffinic hydrocarbons. The yield of gasoline of E.P. 400° F. was 36·5%, and the residue was subjected to cracking in an experimental plant at a pressure of 352 lbs. using transfer temperatures of 896/923°F. Yields of cracked gasoline (E.P. 400°F., anti-knock value equivalent to octane numbers of 68/71) ranging from 51·1/62·8%, of residue (visc. Furol at 122°F., 35/129 secs.) from 48·7/31.9% and of gas and loss from 0·2/6·2% were obtained. In this way the products obtained from the Joiner crude were:—gasoline 68·4/75·7%, cracked residue (meeting Bunker C. specification) 30·4/19·9%, gas and loss 1·2/5·0%. A test was also carried out in a commercial plant by the non-residuum method at a pressure of 170 lbs., and transfer temperature of 920°F., using a residue from a distillation of the crude in a commercial plant, yielding 79% of pressure distillate. From this pressure distillate 69% of gasoline having an E.P. of 430°F. and an anti-knock value superior to that of Ethyl Standard Gasoline was obtained. The total yields from the crude obtained by this method of operation were 61·5% of gasoline, 5·9% kerosine, 12·0% light gas oil, 9·7% pressure distillate bottoms, 10·9% gas, coke and loss. R. A. E.

963. Anti-Knock Gasoline from East Texas Crude. F. R. Staley. *Petr. Eng.*, 1931, **2** (10), 18.

One of the largest and most modern plants in E. Texas, having a capacity of 15,000 brls., employs a process developed by G. L. Rowsey and produces a U.S. motor gasoline of high anti-knock value (octane number 65/70). The plant is very compact and simple in design, and all pumping plant is electrically or motor driven. No steam boilers are required, steam for distillation being generated in the bubble tower from a water spray. Three pipe stills are employed, the first two, which are operated in parallel, being used for skimming operations in conjunction with 1 small and 2 large bubble

towers. The small bubble tower removes light vapours, the first large one a gasoline of E.P. 375°F., and the second a gasoline of E.P. 400°F. from the top, and a kerosine of A.P.I. grav. 41° from the side. The residue is passed to the third or high-pressure pipe still, where it is heated to 860°F., and fractionated in a third large bubble tower from which U.S. motor gasoline of octane number 65/70, and a gas oil meeting U.G.I. specification are obtained. Vapours from the towers are condensed in Bentube sections, and no receiving house is used, automatic control instruments being employed on fractionating towers and to record vapour line temperatures. The straight-run gasoline is non-corrosive and colour stable and requires no chemical treatment. It possesses an anti-knock value equivalent to an octane number of 55/65, depending upon the E.P. The kerosine is also water white and sweet, but the pressure gasoline requires treatment with sodium plumbite.

Employing crude from the Joiner, Bateman and Lathrop fields, this method of operation yields approx. 45% of gasoline, 6% of kerosine, 26% of gas oil, 20% of fuel oil and 3% of loss.

East Texas crude is a mixed base oil and is very desirable from the refining standpoint, yielding a large percentage of straight-run gasoline and being amenable to treatment in a variety of ways. Considerable quantities of paraffin wax and lubricating oils are obtainable if desired, and consequently the crude is being utilised in refineries outside East Texas. Due to the high wax content the topped residue usually has a pour-point of 90/100°F.

R. A. E.

See also Abstracts Nos. 965, 1027, 1030.

Gas.

964. Cracking of Gases on Commercial Scale. W. J. Podbielniak, *Oil and Gas J.*, 1931, **29** (52), 22.

The present surplus of natural gas and waste refinery gases has drawn attention to the production from them of aromatics and unsaturated hydrocarbons by cracking. There are no published accounts of any successful commercial processes of this type, though much experimental work has been done.

The present investigation was undertaken to examine the possibility of commercial cracking of stabiliser vent gas, and possibly natural gas, and to develop suitable equipment and processes for this work.

It has been shown that above 1,000° C. all hydrocarbons in time can be cracked to carbon, hydrogen and methane, while above 1,200° C. methane can be cracked. To obtain higher hydrocarbons as products the temperature, pressure and time of reaction must be controlled very closely. No successful catalysts have been found to reduce the temperature of reaction. A continuous tube type furnace has been designed, the furnace containing a plurality of burners to enable accurate control of temperature in all parts of the furnace to be kept. The capacity of the furnace is 25,000 c. ft./day per coil. A description and views of the experimental plant is given.

A series of runs was carried out, varying only one of the variables and keeping the remainder constant in order to arrive at the optimum operating conditions, the cracked gases being compressed and the aromatic condensate withdrawn and examined.

Tables of the data on the properties of the cracked gases and condensate are given.

It is shown that the maximum yields of aromatics are given by cracking between 1600/1700° F. and of ethylene between 1500/1550° F.

The condensates contained 75/85 per cent. of motor spirit which contained 30/60 per cent. benzole, 15/30 per cent. toluole and had a higher anti-knock value than pure benzole (equal to 1·5 times the volume of benzole). C. L. G.

965. Solubility of Natural Gas in Crude Oil. B. E. Lindsly. *Oil and Gas J.*, 1931, **29** (52), 92.

Samples from the well head were obtained in suitable bombs at existing well head pressures from flowing wells and the amount of gas liberated on gradual reduction of pressure measured and the gas analysed. Samples were obtained at a pressure as high as 1686 lb./sq. in. absolute. A description of the apparatus employed for taking samples and measuring the amount of gas liberated at reduced pressures is given. Allowances were made for presence of water in the gas in the measuring bottles, volume of separator and tubing, and for shrinkage of oil due to liberation of gas. The gas volumes were reduced to a temperature base of 70° F. and a pressure of 14.4 lb./sq. in. absolute. The results show that a much greater volume of gas is liberated by reducing the pressure of the well head sample than has been anticipated from the work of previous authors who have employed the method of measuring the volume of gas absorbed by weathered crude oil when the two substances are contacted under pressure. The volumes of gas liberated by samples of crude oil from Kettleman Hills, California, and Oklahoma at varying pressures are shown in graphical form to illustrate maximum and minimum results obtained. The curves are concave, showing that solubility is not a linear function of pressure. It is thought that the laws of Henry and of Dalton are not strictly applicable when dealing with large concentrations of gas, also that selective solubility is a factor which must be taken into account. More of the heavier gases are absorbed in the oil, *e.g.*, one crude at 25 lbs. gauge pressure absorbed 160 cu. ft. propane, 30 cu. ft. of ethane and 5 cu. ft. of dry natural gas per brl. of oil. Applying this theory to a hypothetical case, curves have been drawn which bear a close resemblance to those prepared from actual tests. It is considered that in natural oil reservoirs as much of the gas as temperature, pressure and gas-oil ratio will allow under conditions of equilibrium has long ago been absorbed and any excess gas is associated with, but not dissolved in, the oil. Under the testing conditions used the compositions of both gas and oil are constantly changing with change in pressure, so a straight line relationship between solubility and pressure is not to be expected. The methods employed by previous investigators did not permit the progressive action of selective solubility as fresh samples were used at each pressure point. Curves are shown to illustrate the shrinkage in oil volume caused by the liberation of gas with drop in pressure. For a Kettleman Hill sample taken at a pressure of 1672 lbs./sq. in. the decrease on reduction to atmospheric pressure was about 37%. This gives an indication of the reduction in liquid content of oil reservoirs caused by liberation of gas, but the full significance of the effect on decline and ultimate production of wells has not been determined. Equal volumes of gas do not result in equal volumetric changes in the oil, the lighter gases causing a shrinkage of about one-third of that caused by the heavier gases. An analysis was made of gas believed not to be held in solution at well head pressure. The volume of this gas was estimated to be 95·4% of the total gas and the natural gasoline content was 0·737 gal. per 1000 cu. ft. The A.P.I. gravs. of the crudes recovered after the experiments are higher than those normally reported by producers,

probably due to retention of propane, butane, etc., usually lost when pressure is suddenly released. This indicates possibilities in improving production methods. Solubilities existing underground probably differ from those at the well-head owing to the differences in pressure and temperature.
R. A. E.

966. Odorization of Natural Gas Effective. R. L. Hamilton and J. T. Rutherford. *Oil and Gas J.*, 1931, **30** (3), 125.

The additions of odorants to natural gas, particularly when used for domestic purposes, is essential to detect leaks which may result in distribution losses and are a source of explosions and toxic dangers to the consumer. The odorant must have a distinctive penetrating odour, but should be non-toxic, non-corrosive, insoluble in water and must burn to harmless and odourless products. A satisfactory odorant, known as Calodorant No. 3, has been developed by the Standard Oil Co. It is a mixture of organic compounds of boiling range 110° F. to 430° F. and contains 4·5% of sulphur, consisting essentially of sulphides. Disulphides and mercaptans are also present. Two and a half to 4 gallons of odorant per million cu. ft. of gas are used. Descriptions of various automatic devices for introducing the odorant are given, together with a table of comparative tests with various odorants for natural gas.
C. L. G.

967. Cooling and Conditioning of Air offers new Uses for Natural Gas. E. D. Milener. *Oil and Gas J.*, 1931, **30** (6), 66.

A new field for the utilisation of natural gas, and one in which it does not compete with or replace another fuel, has been opened by research on its application to house cooling and summer air conditioning. This will assist in balancing the load factor of gas distribution systems, many of which carry a very high load in winter for heating purposes and a comparatively low load in summer. The research has passed the laboratory and early development stages, and three typical houses are to be air conditioned by the newly developed units this summer under the supervision of the committee on industrial gas research. A general discussion on ventilation methods, and on temperature and humidity requirements, follows. The two factors which need to be positively controlled by air conditioning plants are dry bulb temperature and moisture content. In the gas plants the former is controlled by evaporative or indirect cooling using water, or by refrigeration, and the latter by passing the correct amount of air through beds of silica gel. The air in the conditioned area is recirculated, only sufficient fresh air being added to meet the needs of the population in that area. The air drying takes place at ordinary temperatures so that the cooling effect necessary is only that required to remove the sensible heat originating in that area. In this way the method of operation differs fundamentally from power operated systems in which the processes of cooling and dehumidifying are generally effected in one step. The defects and limitations of this method of operating are pointed out. It is anticipated that as the new system will be suitable for home use as well as for industrial application, an increased summer demand for natural gas will be occasioned.
R. A. E.

968. Natural Gas supplied for Manufacture of Dry Ice. K. C. Sclater. *Petr. Eng.*, 1931, **2** (8), 35.

Solid carbon dioxide is now being manufactured in large quantities from the natural gas from an oil well south of Tampico and also from a well in Walden, Colorado. (A description of the "dry ice" plant in Mexico is given in *Ind. and Eng. Chem.*, 1931, **23**, 256.)

Manufacture is effected in three stages: (1) Purification of the gas by chemical means or by scrubbing; (2) liquefaction by compression; (3) rapid evaporation of the liquefied gas to produce CO_2 snow. For a high snow yield the liquid should be cooled to at least —30° F. It is expanded through a fine nozzle into the snow chamber. One type of expansion chamber consists of a cylindrical double-walled chamber 5 ft. long and 3 ft. in diameter. The inner chamber is heavily lagged and maintained at a pressure slightly above atmospheric. About 60% of the cooled liquid is converted to snow in this chamber by sublimation of the gas cooled by the rapid evaporation, while a small percentage is retained in the pores of the snow. The remainder is circulated round the expansion chamber or used for cooling the liquid CO_2. The snow is removed from the chamber and compressed into blocks usually 10 in. square, weighing 40 lbs., being finally packed in special heavy paper for shipping.

A desirable feature of solid carbon dioxide is its slow rate of evaporation, which is attributed to the gas filled voids which make up about 25% of its volume and form an insulating layer of cold gas. The heat conductivity for this reason is much lower than that of a solid block of ice. As a refrigerant, 1 lb. of solid carbon dioxide is almost as efficient as 14 lbs. of solid ice, while it is very effective as a rapid cooling agent owing to its evaporation temperature (in CO_2 vapour —112° F. and in air —137° F.). Another advantage of dry ice as a refrigerant is the absence of moisture, evaporation leaving no residue, thus promoting cleanliness and obviating the destructive effect of corrosion and decay. C. L. G.

See also Abstract No. 969.

Motor Fuels.

969. Operators' Problems in Rectification. The Factors Affecting the Production of Standard Grades of Natural Gasoline. N. K. Rector. *Oil and Gas J.*, 1931, **29** (47), 34.

From a knowledge of the percentage composition of the feed and of the final product having the desired gravity, vapour pressure and recovery, it is possible to deduce the following information by applying Raoult's law and the known vapour pressures of hydrocarbons at various temperatures. (1) The temperature/pressure relationship required in the reboiler; (2) the operating pressure necessary to condense the reflux to the fractionating column. Illustrations of the method of calculation are given, and the function of each part of the equipment outlined. The temperature of the feed to the fractionating column should be that at which the vapour pressure of the mixture is equal to the column pressure and the feed should be introduced at the tray on which the temperature is nearest to that of the feed. The effects of column capacity, butane content of the reflux, reboiler temperature and heater surface, on the operating pressures are discussed. The necessity for constant operating conditions is stressed, and the employment of surge tanks having a storage capacity for at least 8 hrs. production between the absorption plant and rectifier is recommended. Changes in the operation of the absorption plant should be closely watched, so that the operation of the rectifier can be altered to suit the requirements of the changed feed, or the final product may not meet the required specification owing to inclusion

of propane. This is indicated by a reduction in the top temperature of the rectifier, and an increase in reboiler temperature is necessary to free the final product from propane.

A gasoline containing propane having the same vapour pressure at the column temperature as one which is propane-free, will have a higher vapour pressure at 100° F., and a lower distillation recovery, and may therefore fall into a lower grade. Losses in plant output and in the total value of the gasoline extracted per day will therefore arise. The minimum amount of automatic controls for efficient operation are: (1) Reboiler temperature controller; (2) a sensitive back pressure regulator; (3) a feed flow controller; (4) reflux flow controller; (5) top column recording thermometer.

Should the distillation recovery of the final product be higher than necessary, an indication is given by the top column temperature exceeding greatly the temperature at which propane has a vapour pressure equal to that of the column pressure.

If it is found that the distillation recovery of the gasoline obtained is too low, the top temperature may show that nothing heavier than propane is being obtained overhead. This indicates a shortage of butanes in the feed and necessitates increase in the reboiler temperature so as to give a top column temperature just over that at which propane has a vapour pressure equal to the column pressure. R. A. E.

970. Modern Developments in Motor Fuels. G. G. Brown. *Oil and Gas J.*, 1931, **30** (1), 70.

The petroleum industry is tending to change its marketing programme from the present three grades: (1) standard motor fuel; (2) premium fuel of high anti-knock value; (3) premium fuel of high volatility similar to aviation grade; to the following: (1) A subgrade sold at a lower price than the standard; (2) standard grade; (3) a premium grade of high anti-knock value and effective volatility adapted to car requirements rather than aviation service. A graph is presented to show for the years 1900—1930, with certain estimates to 1934, the average 10%, 50% and 90% distillation temperatures of gasolines sold in the U.S.A., their average price and estimated octane number, the total production of gasoline, the registration of motor vehicles, and the production of cracked and natural gasolines. The effects which economic forces, specifications prepared by government committees, and research work have had on the quality of the motor fuels marketed are discussed. The importance of changing motor fuel volatility to suit atmospheric conditions is now recognised, but the changes effected are not adequate to compensate for the temperature differences experienced. The relative ease of starting and acceleration, etc., in the average type of car of the average gasolines sold in selected years during the period 1900—1930 is shown on a graph which also permits calculation of the increase in volatility at salient distillation temperatures necessary to give equal performance at different atmospheric temperatures. Practically all the increase in volatility which has taken place since 1918 is in the first half of the distillation curve. A.S.T.M. committees are engaged in the task of relating test data and engine performance in order to evaluate gum, corrosion and vapour lock and their findings will probably be incorporated in a general specification for gasoline, in which the importance of seasonal volatility change will doubtless be recognised. There is a tendency to increase the anti-knock value of standard grades to an octane number of 70, but whether this figure will become general or not depends on economic considerations and future engine design. Generally speaking, at present the average maximum economic anti-knock value of cracked gasoline is 70/75 octane number, a higher value entailing

high loss and low yield. The present average motor fuel has an octane number of 59/60. The increase in anti-knock value obtained by adding natural gasoline to blending stock is greater than that calculated arithmetically from the known octane numbers of the two gasolines, especially when using cracked blending stock. The importance of natural gasoline for improving anti-knock value will, however, tend to diminish and its main function will be that of improving effective volatility, consequently the vapour pressure should be about 15/20 lb. (Reid) in order to permit inclusion of 30—50%. R. A. E.

971. Effect of New Grading on Production (of Natural Gasoline). R. C. Alden. *Oil and Gas J.*, 1931, **30** (1), 74.

A recent survey of the products from the gasoline plants of a large company has shown that 95% fall in ten grades covering the range up to 30 lb. vapour pressure, and it is thought that 90% of the total production will be confined to thirteen grades (see Abstract No. 183). The seven principal grades will be 30—70, 26—70, 22—55, 22—70, 18—55, 14—55 and 14—40, whilst grades 30—55, 26—55, 22—40, 18—40, 18—70 and 14—25 will be generally available. It is probable that increased production and conservation of light hydrocarbons at refineries will create a demand principally for natural gasolines of relatively low vapour pressure.

A chart is presented showing the relationship between butane and pentane ratio on the one hand and vapour pressure and recovery at 140° F. of the gasoline on the other. This type of chart may be utilised for any given plant to determine its efficiency, which is indicated by the curvature of the line. The pentane ratio of the gasoline will determine the location of the curve and limit the grades which can be readily manufactured. The chart also demonstrates that vapour pressure will be used as the controlling factor in plant operation, since considerable changes can be effected in the vapour pressure of the plant output without altering the recovery at 140° F., and without involving such a large manufacturing loss as when controlling the recovery at 140° F. It is shown that volume losses for vapour pressure changes are almost independent of the pentane ratio for propane-free gasolines.

From a consideration of the losses involved in changing from one grade to another, tables are prepared to show the relative price ratios which gasolines having vapour pressures of 10-30 lb. must command in order to equalise the plant income. Another chart is given to show the A.P.I. gravity which Mid-Continent natural gasolines falling within the thirteen grades will have when prepared on plants having reasonably efficient fractionating apparatus. An important point regarding the new grading system is that it is designed to meet average blending conditions, when approximately 15% of natural gasoline is used. When larger percentages are employed it has been found necessary to specify the vapour pressure by increments of 1 lb. and a closer specification of the recovery at 140° F. is also desirable though not so important. R. A. E.

972. Photographic Flame Studies in the Gasoline Engine. L. Withrow and T. A. Boyd. *Ind. Eng. Chem.*, 1931, **23**, 539-547.

Several investigators have studied photographically the processes of combustion in closed vessels of pinking and non-pinking fuel-air mixtures. This paper describes the apparatus and method by means of which the explosions in an actual engine have been recorded, and the difference between the two types of explosion made clear.

The engine, a single cylinder side-by-side valve unit, was fitted with a special head in which was mounted a quartz window extending the full width of the ceiling of the combustion chamber. The method of mounting the window in the tapered Invar retainer is described in detail. Simultaneously with the flame photographs, records of the pressure fluctuations were made by means of an electrical indicator and single element oscillograph. The flame records were similar to those obtained by Maxwell and Wheeler for explosions in closed bombs, showing that, when pinking occurs, there is a marked increase in the rate of flame movement and pressure rise within the last portion of the charge to burn. In some instances the records show that spontaneous ignition, ahead of the initial flame, occurred in the pinking explosions. Other records show a faint luminosity in the unburned portion of the charge for a short period before the knock began. G. B. M.

973. Contribution to the Study of Mixtures of Petrol-Alcohol-Benzole. C. Baron, C. Boulanger and R. Le Grain. *Rev. Petrolifere*, 1931 (428), 762.

The tendency for moisture to separate in the carburettor from petrol-benzole mixtures as used in aircraft engines, is completely suppressed by the addition of alcohol. The behaviour of petrol-benzole mixtures when cooled to −45° C. was studied and a curve is given showing that the stability decreases as the proportion of benzole increases—a mixture containing 25% of benzole being unstable at −5° C. The petrol used has sp. gr. 0·698, F.B.P. 115°. With petrol-alcohol (99°) mixtures, however, the stability increases as the proportion of alcohol increases. A table shows the temperatures at which trouble occurs with mixtures of petrol-benzole-alcohol. 10% of alcohol added to 80% petrol, 10% benzole; 70% petrol, 20% benzole; and 60% petrol and 30% benzole, gave temperatures at which separation occurred of −27° C., −30° C. and −43° C. respectively.

The addition of alcohol not only lowers this temperature, but also apparently prevents the crystallisation of certain components of the benzole.

It is concluded that the most satisfactory mixture is 70% aviation petrol, 20% benzole, and 10% absolute alcohol. C. C.

974. Heat Treatment of Gasoline. Sir H. W. A. Deterding. E. P. 349,812, June 4th, 1931. Appl., June 27th, 1930.

Gasoline fractions obtained by the straight distillation of petroleum are subjected to a heat treatment under cracking conditions preferably under pressure. The gasoline is first separated by simple distillation into a lighter and a heavier fraction and the latter is subjected to a heat treatment. The treated fraction is then united with the untreated fraction or with some corresponding fraction from another source. Nickel, cobalt, vanadium or silica gel may be added to catalyse the conversion. W. S. E. C.

See also Abstracts Nos. 997, 1000, 1028.

Kerosine.

975. Hydrocarbon Oil of Good Non-smoke Rating. C. I. Robinson, Assr. to Standard Oil Development Co. U.S.P. 1,809,183. 9.6.31. Appl., 10.11.22.

Paraffin in the solid or melted state from petroleum, shale oils or low temperature carbonisation plants is charged into a still and subjected to cracking or pyrolytic decomposition at a temperature of 675° to 750° F., and if pressure is applied it must not exceed 60 lb./sq. in. The overhead

distillate obtained is separated into naphtha and refined oil. The refined oil is a very superior burning oil, it has a very high gravity (50–54° B6), a distillation range between 300° and 575° F., a low sulphur-content and a high non-smoke tendency as determined for Pennsylvanian kerosines. Since the oil burns without forming wick-crusts it is specially applicable for use in signal lamps. W. S. E. C.

See also Abstract No. 997.

Lubricants and Waxes.

976. Recent Developments in Contact Filtration. G. Pfau and C. A. Barrere. *Petr. Eng.*, 1931, **2** (9), 140.

A brief description is given of the contact process of treating acid refined lubricating oils with dry clay in order to neutralise the oil and bring it to the desired final colour. The types of natural and chemically treated clays used are mentioned, and methods for treating the clays are given. The efficiency of a clay must be determined by laboratory examination, as a chemical analysis is not a satisfactory criterion. Where the clay is used in the dry state it must be finely ground (90% through 200 mesh). Decolorising action generally increases with increase in temperature, the usual range for contacting being 400-500° F. A closed continuous system is used to prevent exposure of hot oil to the air.

Clay may also be used in pulp form, in which case it must be prepared at the point of usage. The raw clay is ground to 75% passing 200 mesh and treated with 50% of its weight of 20% acid, the clay being slowly added to the boiling acid with air and steam agitation. When the temperature reaches 220-230° F. agitation is discontinued and the mass allowed to digest for 12 hours. Sludge and acid are separated and the acid may be recovered for future treatments. A loss of 25-40% of clay is usual. The pulp is removed to wash tanks and washed three times with water heated to 150° F. for 20 min., settling and drawing off water each time requiring 6-8 hours. The pulp contains about 25% solids, and should not have an acidity in excess of 3 mg. of KOH per gm. The pulp is added to acid oil at the rate of 0·2 to 2 lb. of clay per gal. of oil and agitated in lead-lined mixing tanks for 1–2 hours. The mixture is pumped to a pipe still separator to remove water from the clay, from thence to a tube still heated to 400-420° F., and discharged back to the separator. Oil is drawn off from this return line at the same rate as the feed to the separator from the mixing tank, and passed through a cooler to the press charging tank. The oil is diluted with 50% of naphtha of boiling range 180-400° F. and filter-pressed in a leaf type filter. In order to remove the last traces of clay, which will spoil the quality of the oil and give trouble in the subsequent dewaxing process, a further filtration through a press using leaves precoated with a filter aid is suggested. The advantages of the method are: (1) Accurate control of final colour of oil, giving a more uniform product; (2) elimination of usual neutralisation troubles; (3) low first cost, greater simplicity of operation and increased flexibility; (4) improved quality of oil as measured by laboratory tests. The clay may be reclaimed by burning at 750-1000° F. or recovered by solvents. Corrosion of plant, which is liable to occur prior to contacting the oil and clay, and the subsequent erosive effects are detailed, and methods for combating the consequent plant depreciation are given. A flow sheet of the whole process is provided.

R. A. E.

977. Progress in Manufacture of Lead-Containing Lubricants. F. C. Otto. *Petr. Eng.*, 1931, **2** (10), 75.

The demand for lead-containing greases is rapidly increasing, particularly for automobile lubrication. Their popularity is due mainly to: (*a*) Their pressure-resisting properties; (*b*) their ease of flow at low temperatures, lower even than the pour point of the mineral oils with which they are blended; (*c*) their high water resistivity; (*d*) the large amount of grease that can be added to an oil to give the required consistency, thus improving the resistance of the grease to pressure and friction. There are three main types of lead-containing greases: (1) Mono-saponified; (2) twofold saponified or double decomposition products; (3) lead-loaded products. The mono-saponified type is more popular than the twofold, owing to the fact that the lead soap tends to precipitate from the latter. Great variation of the melting point of the finished grease can be obtained owing to the wide range of the m. pts. of the various lead soaps (from 113-122° F. for lead oleate to 257-260° F. for lead stearate).

Details of the manufacture of mono-saponified and double decomposition lead soap greasers are given. The former consists essentially of the manufacture of the lead soap (usually from lead oxide or lead carbonate and whale oil or oleic acid) and its dilution with mineral oil to the required consistency.

Twofold saponified greases almost invariably consist of lead oleate, which is prepared by treating oleic acid with sodium or potassium hydroxide and reacting with lead acetate, sodium acetate being decanted off.

Lead base lubricants are particularly in demand in the automobile industry, owing to their unctuous free-flowing nature. The lead tends to go into intimate connection (possibly of a chemical nature) with the steel surface lubricated. Practical tests have shown that sodium and calcium base lubricants will resist in rear end drives a teeth pressure of only 8000 lb., while lead base greases will stand more than 25,000 lb. A disadvantage of lead base greases is that absorbed moisture causes precipitation of the lead soap. They should therefore always be stored in closed containers. Some grades of lead base greases on the market have failed owing to their being prepared from cheap black oils of insufficient viscosity. Asphaltic base bright stocks of viscosity greater than 150 at 210° F. are suitable for blending. Precipitation of the lead soap can also be avoided by the selection of a suitable fatty oil, the soap of which will remain in suspension in the particular mineral oil being used. The third type-lead filled greases are used on account of their good pressure-resisting qualities for heavy rough work. In one case 30% of white lead and 30% of graphite are added as fillers. Lead dust and babbit metal dust are also used. C. L. G.

978. Distillation of Lubricating Oils in the Cathode Vacuum. Louis and A. Chmelevsky. *Ann. des Comb. Liquides*, 1931, **6**, 59–74.

The authors have modified Iselin's apparatus for the distillation of oils in the cathode ray vacuum and used it for the comparison of results obtained by this method and employing 14 mm. pressure. Five oils from Russian and Pechelbronn crude were fractionated and the results tabulated.

Better fractionation was obtained with the higher vacuum, the distillates contained no deposits and had no "cracked" odour, both these defects applying to the oils distilled at 14 mm. The fractions distilled at 14 mm. are also more easily alterable (in Woog's test).

The method of distilling in the cathode vacuum permits the production of oils of high viscosity; it might also be useful for isolating constituents of oils. E. B. E.

979. Synthetic Lubricating Oils. F. W. Sullivan, B. Voorhees, A. W. Deeley and R. V. Shankland. *Ind. Eng. Chem.*, 1931, **23** (6), 604–610.

A systematic investigation of lubricating oils made by the polymerisation of olefines with aluminium chloride has been carried out. Various pure olefines up to C_{16}, including all the isomeric C_4 and C_5 mono-olefines and cyclic olefines, as well as unsaturated distillates from cracking various materials have been investigated. The following conclusions have been reached: (1) The temp. coeff. of viscosity of the polymers decreases with increasing mol. wt. of normal olefines and increases with increasing branching of the chains for isomers (range covered corresponds to a viscosity index of 140 to less than −300); (2) the polymerised oils from the cracked distillates were paraffinic to a degree corresponding to the wax content of the charging stock (polymers of the higher fractions superior to those of the lower boiling cuts); (3) the product is wax-free although mainly paraffinic; (4) polymerised oils cracked and repolymerised give oils becoming successively poorer with regard to temp. coeff.; (5) polymers of cyclohexene—particularly turpentine and cracked distillates from naphthenic charging stocks—are more susceptible to change in viscosity with temp. than natural oils. Various deductions are made of possible types of constitution for lubricating oils.

A commercial process has been developed for the production of oils by this means. The products are well suited for uses where constancy of viscosity with varying temp. or high oxidation stability are required. W. D. S.

980. Composition and Crystal Form of Petroleum Waxes. S. W. Ferris, H. C. Cowles and L. M. Henderson. *Ind. Eng. Chem.*, 1931, **23** (6), 681–688.

The physical properties and crystal forms of so highly purified paraffin wax fractions from a Mid-Continent petroleum have been examined. The results indicate that petroleum wax consists of several hydrocarbon series which are homologous at least with respect to physical property and crystal form. The members of a given series show marked similarity in crystal form. Three types, plate, needle and malcrystalline have been distinguished; the plate type exhibits for a given b.p., the highest m.p., and the needle the lowest. The crystal form is an inherent property of the compound and is independent of factors such as viscosity of the solvent. Change in the chemical character of the solvent may change the shape but not the general characteristics of the plate. Both needle and malcrystalline waxes have marked powers of impressing their form upon plate waxes, the malcrystalline form apparently being more powerful in this respect than the needle. However, neither type of wax can affect the form of wax plate unless the solubility relationships are such that the needle or malcrystalline waxes separate from solution simultaneously with the plates. W. D. S.

981. X-Ray Diffraction Study of Paraffin Waxes. G. L. Clark and H. A. Smith. *Ind. Eng. Chem.*, 1931, **23** (6), 697–701.

An X-ray diffraction study of fractionated paraffin wax samples proves that no fraction can be a pure compound, and verifies the conclusion that paraffin wax is composed only of *n*- and *iso*-paraffinic hydrocarbons. Two normal paraffin molecules $C_{28}H_{58}$ and $C_{41}H_{86}$ have been identified for the first time. It is shown that the X-ray diffraction method can be of considerable aid in evaluating the amounts of *n*- or *iso*-paraffin molecules present in crude waxes. Of particular interest are the diffraction patterns which show that the higher the m.p. the greater the number of orders for the identity period corresponding to molecular length, indicating more perfect orientation. Only 5 different hydrocarbons—*i.e.*, those with 29, 31, 34, 38 and 42 C. atoms—impress their own spacings on the sixteen fractions examined. W. D. S.

982. Production of Lubricants. Standard Oil Development Co. E.P. 348,834, 21.5.31. Appl., 11.4.30. And E.P. 349,342, 28.5.31. Appl., 19.6.30.

High grade lubricants are produced from heavy hydrocarbon oil after all non-crystalline impurities of a gummy, resinous or asphaltic nature have been removed. The purified oil is hydrogenated at pressures above 100 atm. and a temperature exceeding 700° F. The second patent describes the production of a high grade oil without the removal of the impurities. An apparatus is described. The conditions of temperature and pressure are 700°–850° F. and above 200 atm. respectively. The oils produced boiling below 400° F. are limited to less than 20% of the oil treated. W. D. S.

983. Production of Lubricating Oils. I. G. Farbenind. A.-G. E.P. 349,071, 11.5.31. Appl., 18.8.30.

Agents capable of introducing exchangeable substituents—*e.g.*, a halogenating agent such as chlorine—are allowed to react on solid or semi-solid hydrocarbons having open chain structure, being rich in hydrogen and having no or only slight lubricating properties. Other desirable properties of the hydrocarbon material utilised are a mean mol. wt. of at least 250 and 13·5–15 parts of H_2 for each 100 parts of carbon. The exchangeable substituents (hydrogen chloride) are split off from the treated products and the resulting unsaturated hydrocarbons condensed, preferably in the presence of cyclic hydrocarbons and/or olefines, by means of aluminium chloride. W. D. S.

984. Vacuum Distillation. W. B. Logan and Texas Co. U.S.P. 1,804,941, 12/5/31. Appl., 27/12/27.

A process for distilling mineral lubricating oil continuously at a pressure of 25–50 mm. of mercury is described. The oil is vaporised in a still in the presence of steam, and the vapours passed up a tower down which water drops. The vapours are condensed and the water evaporated. The condensate comprising the desired lubricating oil is run off from the base of the tower. W. D. S.

985. Treating Oils. W. M. Stratford, Assr. by Mesne Assignments to the Texas Co. U.S.P. 1,807,183, 26.5.31. Appl., 24.11.24.

Lubricating oil is agitated with sulphuric acid in two portions, the first constituting not more than one-third of the total quantity of acid used, and the acid and sludge products are coagulated after each acid treatment by agitating the mixture of acid and oil with a comminuted solid material. The oil is neutralised after the second acid treatment and coagulated by mixing with an alkaline solution and passing steam into the mixture. W. S. E. C.

986. Dewaxing Mineral Oils. L. Edeleanu. U.S.P. 1,809,777, 9.6.31. Appl., 11.1.30.

A process for dewaxing minerals oils is described. The upper layer liquid obtained from the Edeleanu refining process is mixed while still saturated with liquid SO_2 with a further excess of liquid SO_2. The temperature is lowered to that desired by decreasing the pressure to cause vaporisation of liquid SO_2 in contact with the oil. The wax precipitated is removed mechanically. It is claimed that the insolubility of the wax or paraffin is increased by utilisation of this method. W. D. S.

See also Abstracts Nos. 987, 1029.

Fuel Oil.

987. Methods of Mixing Sludge Fuel. G. Pfau and C. A. Barrere. *Petr. Eng.*, 1931, **2** (10), 129.

Treatment of heavy acid sludge to render it suitable for combustion is most conveniently carried out by the two stage method, usually described as the "cutter" and "body" acid treatments. Acid sludge should never be deposited on the ground in the open, owing to its susceptibility to oxidation, rendering it impossible to treat or burn successfully. Conversion of acid sludge to fuel is carried out in lead-lined kettles with conical bottoms.

A description of a suitable apparatus with full details of the procedure is given. The process consists of agitating with steam and air, settling, adding 30% of fuel oil and extracting the acid with hot water in two stages. The temperature of the mix should not exceed 210° F. to prevent coking of the asphaltic particles. The acid water removed in the first stage has a strength of 18–35% sulphuric acid, and in the second 10–15%. In a typical test, 74·2% of the total acid was recovered, the resulting sludge fuel being homogeneous, free from lumps and having no tendency to settle out.

Treatment can be carried out in one stage, but owing to the difficulty of separating the acid water a 40% fuel mix is usually the limit obtainable. If there is no possible use for the acid water the sludge fuel can be mixed with an equal quantity of heavy fuel (200–300 secs. at 162° F.) and burnt, preferably neutralising with soda ash, etc. The addition of petrolatum from dewaxing plants in greater proportion than 15% is not successful unless the acid has been first removed from the sludge. Where light and heavy sludges are produced it is advantageous to work them up together, owing to greater ease of handling the heavy sludge. The use of soda ash for neutralising usually results in foaming in the kettles, plugging of the burner tip and scale on boiler or still tubes.

Corrosion troubles can be met by using brass or bronze lines, valves, etc., while if lead lines are used they should contain 10% antimony. With an acidity of less than 1% there should be no evidence of corrosion. Any ordinary fuel oil burner may be used, but the tip should be ¼ in. or larger, while steam atomisation should be employed generously. The sludge fuel should be kept in constant agitation by air and heated with steam coils.

The experience of the authors is that boiler firemen prefer to burn a smooth, hot, homogeneous sludge fuel, rather than a straight fuel oil.

C. L. G.

Fluxes and Asphalts.

988. Emulsions of Tar. N. Bendixen and J. G. Y. D. Morgan. E.P. 346,978, 23.4.31. Appl. 24.4.30.

This patent deals with the manufacture of aqueous emulsions of tar for use in the surface dressing of roads or pavements. The emulsions are produced by agitating the tar with a dilute aqueous solution of the saccharate formed from a mono- or di-saccharide in a quantity not exceeding 10% by weight of the tar. Dilution with xylol, tetrachloracetylene, carbon tetrachloride, etc., increases the drying properties of the emulsion. The character of the final product can be varied by using different saccharates and in varying proportions.

W. S. E. C.

Special Products.

989. Conversion of Butylenes into Liquid Hydrocarbons. A. Mailhe and —. Renaudie. *Ann. des Comb. Liquides*, 1931, **6**, 79–81.

Ethylene and propylene, on passing over heated silica gel, have been shown previously to yield liquid products. The three butylenes have now been examined similarly.

Butene-1, on passing through a tube containing silica gel at 650–670° C. yields an oil which condenses on leaving the tube, and spirit which is absorbed by active carbon. The liquid products amount to 33% of the butene reacted; the spirit contains about 21% of aromatic hydrocarbons, principally benzene and toluene. The uncondensed gas contains unsaturated hydrocarbons together with about 38% methane. Methyl propene gives similar results under these conditions. Benzene, toluene and m-xylene were identified.

Butene-2 was examined at 700°. It yielded 37·7% of liquid products, the spirit containing 55% aromatics. Higher working-temperatures favour the production of cyclic hydrocarbons. E. B. E.

990. Substitute for Toluole in Cellulose Lacquer Formulation. *Oil and Colour Trades J.*, 1931, **79**, 1607.

The superiority of toluole as a diluent in cellulose lacquers, together with its high price, have stimulated searches for a substitute. In America, a petroleum naphtha called "V.M.P. Naphtha" (Varnish Makers' and Painters' Naphtha) has an extensive use in place of toluole. Judicious variation of solvents in a formula has shown that toluole could be partially replaced by the naphtha with a very small loss in solvent power and a definite saving in cost.

In England work has been done with a petroleum naphtha available in large quantities, and having the following properties:—Boiling range, 100–140° C.; aromatic content, 40% (I.P.T. Method). The article gives results of work done to determine the tolerances for toluole and petroleum naphtha and, for comparison, results are quoted from the American literature dealing with "V.M.P. Naphtha." The tolerance figure is the volume in mls. of the diluent which can be added to 20 ml. of a solution of nitrocotton in a solvent before precipitation of the nitrocotton occurs. The solution is so adjusted as to give a final concentration of about 8%. The tolerance for both toluole and V.M.P. Naphtha varies with the solvent (ethyl acetate, etc.), and while toluole always has a higher tolerance figure than the naphtha, the ratio of the two varies over a fairly wide range. Further work is suggested on the use of a white spirit (18% aromatics). C. I. K.

991. Market for Specialities Expanding. R. C. Conine. *Oil and Gas J.*, 1931, **30** (4), 18.

Reference is made to the very large number of special technical products developed by the S.O.C. of Indiana from petroleum oils, residues, bye-products, etc.

Particular reference is made to insecticides and tree sprays, polishes, cosmetic products, cutting oils, special lubricating oils, special greases, products embodying phenols and naphthenic acids (for wood preservatives, antiseptics, anti-fouling paints, etc.), and to the possibility of producing organic chemicals by chlorination or oxidation of cracked products and waste gases. C. L. G.

992. Manufacture and Production of Unsaturated Hydrocarbons. J. Y. Johnson. E.P. 347,620, 17.4.31. Appl. 17.1.30.

Unsaturated liquid hydrocarbons are manufactured from liquid or solid paraffin or naphthene hydrocarbons, by oxidising these substances and subjecting the products to such high temperatures that water and/or carbon dioxide are removed. A catalyst is used in the reaction and the carboxylic acids are removed by treatment with alkali before increasing the temperature to drive off water or carbon dioxide. W. S. E. C.

993. Manufacture and Production of Polymerisation Products of Diolefines. I.G. Farbenind. A.-G. E.P. 347,802, 7.5.31. Appl. 28.2.30.

The polymerisation of diolefines in the presence of alkali or alkaline earth metals or mixtures of these metals is considerably facilitated by the presence of small amounts of organic saturated or unsaturated halogen compounds. Suitable compounds include ethylene chloride or bromide, 1 : 2-propylene dichloride, vinyl chloride, vinyl bromide, *cis*-dichloro-ethylene, *trans*-dichloro-ethylene, etc. The polymerisation can be effected with good results between 0° and 100° C., but between 15° and 65° C. is to be preferred. S. F. B.

994. Treatment of Saturated Hydrocarbons and Gaseous Mixtures Rich in Saturated Hydrocarbons. Société d'Etudes Scientifiques et d'Enterprises Industrielles. E.P. 349,067, 14.5.31. Appl. 14.1.30.

Saturated hydrocarbons in a gasified or atomised state are admixed with steam and oxygen and passed through a reaction chamber at 1300° C. at a high velocity. It is claimed that considerable amounts of acetylene, carbon monoxide and hydrogen are formed. H. C. R.

See also Abstract No. 968.

Refining and Refinery Plant.

995. Developments in Refining Pennsylvania Crude Oil. C. M. Ridgway. *Petr. Eng.*, 1931, **2** (9), 109.

Pennsylvania crude is now employed principally for producing lubricating oils and is practically uniform in quality. Variations in gravity merely give differences in gasoline and kerosine content, but do not materially affect the quality of the lubricating oil fractions. The crudes are generally processed to yield as the main product either a long residuum of 90 sec. viscosity at 210° F. or a bright stock of 150 sec. viscosity at the same temperature. The normal yield of products obtained by both methods of operation are quoted. The E.P. of the gasoline is now generally maintained at 360—380° F. in order to improve the anti-knock value. Three types of kerosine (A.P.I. grav. 45, 46 and 47 respectively) are produced and are all of excellent quality. The gas oil is now often used as cracking stock to improve the anti-knock value of the gasoline. The wax distillate is used for the production of neutral oils and wax and the normal yield and properties of the products obtained are quoted. The methods employed in dewaxing the steam refined stock are detailed and full specifications of the neutral oils, steam refined stocks, bright stocks and motor oils produced are given in the form of tables. R. A. E.

996. Chemical Treatment of Distillates from Sour Crudes. Pt. 2. Vapour Phase Methods. C. B. Fraught. *Refiner*, 1931, **10**, 70.

Colour, odour, gums and instability of cracked pressure distillate from cracking plants complicate the usual refining treatment, and doctor sweetening alone is not sufficient to yield a marketable product.

Vapour phase treatment has been used successfully for such distillates, and there are several patented processes which make use of a body of clay alone, or mixed with chemicals, but the former is the only process used universally. In this process the clay is placed in a vertical container with a false bottom, below which the polymerised material is withdrawn. The vapours pass through the mass of granular clay into condensers. The clay acts as a catalyst and polymerises the diolefins, converting them into high boiling range hydrocarbons which become liquid at the temperature of the clay and trickle down to the outlet at the lowest part of the tower. This selective polymerisation appears to require definite amounts of catalyst irrespective of the nature of the gasoline and does not appear to be affected by the presence of sulphur compounds.

The rate of flow checks well in the laboratory against works practice, but the life of the clay is much shorter in the laboratory. This seems to indicate that the ability of the clay to free itself from polymers determines its effectiveness.

The rate of throughput for maximum efficiency is 0·6 c.c. of condensed vapours per 1 g. of clay per hr.

Various clays are used and very little difference has been noticed in results from standard 16-30, 30-60 and 60-90 mesh clays.

The total recovery on crude is materially increased, when compared with acid treatment and redistillation, by the use of the vapour phase process. Vapour phase treated pressure distillate has one peculiarity. Unless the finished distillate is negative to the doctor test the colour will revert on storage.

Where less than 0·10% of sulphur is specified in the gasoline, acid treatment and re-running is necessary. All traces of hydrogen sulphide must be removed before acid treatment, otherwise free sulphur is liberated and the finished gasoline will contain more sulphur than the original sample. Pressure distillates darken under treatment and require more care in re-running than straight-run spirit, so it is beneficial to mix the two before acid treatment. Speed is essential in using large quantities of acid as it is easy to "burn" the distillate and make re-running difficult. Continuous acid treatment is difficult as it is necessary to be sure that every barrel of spirit receives its proper dosage. Batch treatment is recommended as follows: Add ½ lb. 66 Bé acid to remove water, draw off. Add the main dump in one increment, agitate for 5 mins., settle for 10 mins. Draw off the acid and wash with 18 Bé soda, settle, run off, and discharge the oil to the re-run tank.

Samples taken from a batch after 5 min. acid treatment show the same sulphur content as a series of samples drawn at 10, 15, 20, 25 and 30 min. agitation. The samples after 5 min. agitation showed the best colour, gum, stability, and odour after re-running. It will be noted that no water was used in this treatment. The use of 8 to 10 lb. of acid is necessary to reduce the sulphur content from 0·25% to below 0·1% and this treatment results in high losses.

Bureau of Mines Bull. No. 333 calls attention to the formation of carbon when acid treated pressure distillate is re-run. This condition gives great trouble when distillates are treated with large amounts of sulphuric acid, and heat exchangers, bubble tower caps and down comers are choked with the carbon-like deposit.

It appears that the use of acid has its limits for the removal of sulphur, and other means of desulphurising are necessary or the allowable amount of sulphur must be raised.

One known chemical which will completely remove sulphur is metallic sodium, and experiments covering a number of years on a commercial scale have been made, but difficulties have not yet been overcome. R. .P

997. Decolorisation and Stabilisation of Distillate Petroleum Products with Respect to Colour and Odour. Standard Oil Co. E.P. 348, 011, 29.4.31. Appl., 29.10.29.

The method described is particularly applicable to refined petroleum products such as gasoline and kerosine which have become discoloured during storage. Various compounds consisting of organic or inorganic reducing agents are added, *e.g.*, one of the following: orcinol, pyrogallol, α or β-naphthol, thiourea, thiocarbanilide, phenyl hydrazine, oxalic acid, stannous oleate, sodium methylate, stannous chloride; these compounds decolorise the oils and prevent rediscoloration. When the reducing agent is a solid it is dissolved first in a solvent which is miscible with the oil to be treated, *e.g.*, ethyl, butyl or isopropyl achohol. The quantity added is less than 0·25% of the volume of oil treated. W. S. E. C.

998. Treating Oil. L. Caldwell, Assr. to Celite Corporation. U.S.P. 1,802,628, 28.4.31. Appl., 29.6.26.

The invention relates particularly to improvements in the refining of petroleum oils, waxes, gums, etc. Oil vapours are brought into intimate contact with diatomaceous silica which has been reacted with an alkaline earth metal hydroxide in the presence of water. W. H. T.

999. Distillation System. A. F. Keane, Assr. to Semet-Solvay Engineering. Corpn. U.S.P. 1,803,671, 5.5.31. Appl., 17.4.26.

This invention claims to effect a saving in labour, water and steam and to afford better control of operation in the separation of light oils from benzolised wash oil.

The apparatus consists primarily of a column made up of four sections, a heat exchanger at the top, a condenser next, thirdly a separator and a liquid collector at the bottom. Vapours from the steam distillation of the wash oil enter the column at the bottom of the heat exchanger section and pass upward over a plurality of coils. These may be arranged in coils connected in series or in banks of tubes and headers in series, and each coil or bank of tubes is separated from the next by a dephlegmator plate and hood. Cold fuel oil is pumped through these coils and is preheated by the hot vapours. From the top of this section, the vapours are carried by an external conduit to the top of the condenser, which is also composed of several sections. Condensate collects on the bottom plate of each section and then overflows to the section below, finally reaching the separator. Non-condensable gas is purged from the system through a purge valve in the lowest condenser section. The lower layer of water is withdrawn from the separator through an adjustable overflow, and the light oils pass to the receiving tanks. W. L.

1000. Desulphurising Cracked Hydrocarbons. G. Egloff, Assr. to Universal Oil Products Co. U.S.P. 1,803,964, 5.5.31. Appl., 26.6.26.

The removal of objectionable sulphur compounds from gasoline is effected by passing the cracked hydrocarbon oil vapours from the dephlegmator into a suitable still maintained at a temperature of 500° F., and subjecting them

to the action of ozone in the presence of a silent electric discharge. The operation can be carried out under vacuum conditions as low as 5 mm. or under a pressure of several hundred lb. per sq. in. W. S. E. C.

1001. Fractionation of Hydrocarbons. C. P. Dubbs and Universal Oil Products Co. U.S.P. 1,804,553–4, 12.5.31. Appl., 14.1.26.

Two pieces of apparatus are described for the rectification of hydrocarbon oil vapours. Both consist of a vertical cylindrical shell with means at the bottom to admit vapours and means at the top for withdrawing uncondensed vapours and gases. In each shell there is a continuous helical baffle made from a thin metal strip of corrugated cross section. The shell described in the first patent has these corrugations parallel and concentric while the spacing of the convolutions formed by the helix decrease towards the top of the shell. The second patent describes a baffle with radial corrugations and perforations in the crests of the corrugations. Both have means for discharging a cooling liquid into the shell from the top. W. D. S.

1002. Producing Low Boiling Point Hydrocarbons. C. P. Dubbs, Assr. to Universal Oil Products Co. U.S.P. 1,806,616, 26.5.31. Appl., 16.9.21. Renewed 16.7.28.

This invention relates to a process for treating hydrocarbon oils to produce therefrom products having lower b. pts. The oil is first pumped through a heating coil and thence to a separating chamber from which vapours are taken off to a vapour chamber. The unvaporised oil is passed through a second heating coil to the vapour chamber, the gases formed being condensed. The unvaporised oil is finally returned to the first coil, the oil being maintained under pressure of the generated vapours during treatment.

W. H. T.

1003. Method of Filtering and Decolourising Oils. D. W. Grant. U.S.P. 1,806, 876, 26.5.31. Appl., 23.6.30.

A process for filtering and decolourising the residual oils from the distillation of crude petroleum is described in which the filter is charged with a body of fuller's earth and kept stationary. The earth is charged with a quantity of oil which is allowed to seep into the earth and to remain in intimate contact with it for a time sufficient to decolorise the oil. The oil is subsequently flushed from the earth with petroleum spirit and the spirit removed from the oil. W. S. E. C.

See also Abstracts Nos. 964, 976.

Cracking.

1004. Removing Carbonaceous Matter from Oil Apparatus. H. W. Sheldon. Assr. Vacuum Oil Co. U.S.P. 1,800,964, 14.4.31. Appl. 11.7.29

The apparatus consists of a number of independent flexible metal cables suspended from various points at the top of the reaction chamber. Each cable has a free end, extending inwardly from its point of attachment to a position near the upper manhole of the chamber, so that each cable may be engaged in turn at this free end and pulled down through the coke and out through the lower manhole. The lower ends of the cables are held in position by hooks at the bottom of the chamber. These hooks open towards the middle of the chamber so that the cables disengage freely when pulled through the lower manhole. W. L.

1005. Treatment of Hydrocarbon Oil with A Metallic Halide. R. W. Hanna, Assr. Standard Oil Co. U.S.P. 1,801,627, 21.4.31. Appl. 27.11.26.

Hydrocarbon oil and anhydrous metallic halide, such as aluminium chloride, are maintained at a conversion temperature and undergo a decomposing reaction in a reaction chamber. This mixture is continuously circulated through a trap which separates the halide sludge. This sludge or fresh halide is returned to the reaction chamber without passing through any heating zone. The separated oil passes through a heating chamber before being reintroduced into the reaction chamber. Vapours evolved in the reaction chamber are refluxed and the condensate from this tower returns directly to the reaction chamber.

Fresh crude is continuously added to the recirculated oil before the latter passes through the heating zone. W. L.

1006. Treating Hydrocarbon Oil with Metallic Halides. R. W. Hanna. Assr. Standard Oil Co. U.S.P. 1,803,670, 5.5.31. Appl. 22.1.27.

The oil and the metallic halide are first mixed and then passed, under a pressure sufficient to prevent volatilization of the unconverted oil, through a heating zone where it is brought to reaction temperature. From here it passes to a reaction chamber where it is held for a short time under the same pressure. While in this chamber, the mixture may be subjected to mechanical agitation and any vapours produced are removed and fractionated, the reflux and condensate from the dephlegmater being returned to the chamber. The mixture passes to a larger chamber through a pressure reducing valve and is here distilled under vacuum. Heat is applied and all the hydrocarbon oil, both converted and unconverted, is vaporised, while the metallic halide sludge is reduced to a coke. The vapours are withdrawn and dephlegmated, the reflux to the fractionating column being charging stock to the plant. Reflux and the condensed unconverted oil from the bottom of the column is recirculated through the plant. Any metallic halide which enters the evaporating chamber without being utilized is there volatilized and is condensed in the column and recirculated with the recycled oil. W. L.

1007. Converting Hydrocarbon Oils. C. P. Dubbs. Assr. Universal Oil Products Co. U.S.P. 1,803,962, 5.5.31. Appl. 1.6.25.

The charging oil is heated to cracking temperature under super-atmospheric temperature and delivered to an enlarged vapour chamber. Combustion gases circulate about the outside of this chamber in spaces provided between the chamber and the outer insulation wall and maintain it at such a temperature that at least 80% of the charging oil is maintained in the vapour phase. The vapours are withdrawn through a swan neck vapour pipe and dephlegmated. Condensate from the dephlegmator is returned to charging stock and recycled while the overhead vapour is condensed and stored. W. L.

1008. Cracking Oils. R. T. Pollock, Assr. Universal Oil Products Co. U.S.P. 1,804,028, 5.5.31. Appl. 14.3.21.

Vapours from the expansion chamber pass out to a valve controlled header. This header is connected to the dephlegmator by a vapour line which is inclined downward toward the dephlegmator. In this vapour line is positioned a spiral baffle plate which may be a helical or a flat baffle. This greatly increases the surface contact of the vapours giving increased condensation and dephlegmation. Condensate formed in this pipe drains into the dephlegmator through spaces provided in the lower edge of the baffle and is withdrawn and recycled with the dephlegmator reflux. W. L.

1009. Process of Generating Power. L. Kirschbraun. U.S.P. 1,804,561, 12.5.31. Appl. 29.12.19.

Oil is raised to cracking temperature by passing through a heating coil, and passed into a vapour chamber, thence through a vapour line to dephlegmator and condensers. In the vapour line between the vapour chamber and the dephlegmator is interposed a turbine, rotary motor, or other type of motive power, which can utilise the expansive energy of the vapours. Means are provided for withdrawing condensate which may form in the motor. Uncondensed vapours are dealt with in the usual type of cooler. R. P.

1010. Cracking Process. W. P. Deppe and L. L. Summers. U.S.P. 1,804,917, 12.5.31. Appl. 8.2.24.

The process consists in cracking heavy hydrocarbons in the liquid film form, in intimate contact with the vapours, so that approximate equilibrium will be maintained between the liquid and the vapours, in contradistinction to prior processes wherein the distilled vapours are removed as fast as they are formed.

In practice the oil is introduced into a retort chamber at the upper end of a column. The liquid is spread over highly heated plates in thin films, then passed as liquid and vapour into a second retort chamber lower down the column. The mixture may be passed through a number of chambers and progressively heat treated at higher temperatures. R. P.

1011. Treating Hydrocarbon Oils. W. K. Holmes. U.S.P. 1,804,933, 12.5.31. Appl. 13.8.25.

In the cracking process there are formed, besides gasoline, polymers which readily decompose to form carbon which tends to concentrate in the plant and limit the continuous nature of the process.

The length of runs may be materially increased by periodically subjecting the converters to a temporary flushing operation. Thus by increasing the rate of charge to the cracking zone and increasing the amount of tar withdrawn the polymers may be removed from the system.

In practice the oil is introduced into a series of converters, the vapours are fractionated and heavy residue continuously removed. The oil in the converters is agitated or stirred during the operation.

When it is observed that the residue in the converters is becoming excessively heavy the plant is flushed by increasing the amount of fresh oil being pumped into the process.

The rate of charge and withdrawal may be so high that cracking is temporarily suspended, but in general it is unusual to conduct such extensive flushing. The frequency of flushing, as well as the extent and character of the same, depend on conditions of operation and the character of the charging stock. Pressure stills may be in continuous operation in this manner for several months at a time. R. P.

1012. Cracking Hydrocarbons. G. H. Taber, Junr. U.S.P. 1,805,113, 12.5.31. Appl. 11.10.28.

In cracking operations in which vapours and gases are subjected to a condensing operation, the improvement consists in subjecting the remaining gases successively to scrubbing treatments with raw charging stock. Two treatments are specified. From the first scrubbing tower the oil is pumped into the upper end of the reflux tower in direct contact with the vapours from the cracking plant. The absorbed constituents are thus recovered without further subjection to a cracking temperature.

The raw stock from the second scrubbing tower is introduced into the cracking still directly, and the absorbed constituents are subjected to cracking temperature in contact with the raw stock. Thus the light hydrocarbons suitable for inclusion in the finished product are separated from intermediate products such as ethylene and related unsaturated components which are returned for further treatment. R. P.

See also Abstracts Nos. 964, 974, 996.

Hydrogenation.

1013. Valuable Products from Coal and Other Carbonaceous Materials. H. D. Elkington, Assr. N. V. de Bataafsche Petr. Mij. E.P. 346,689, 7.4.31. Appl. 30.1.30.

A method of preparing catalysts, to be used in the treatment of coal, tars, mineral oils, etc., with hydrogen or other reducing gases, is described. A mass is obtained by absorbing a colloidal preparation of the catalytic compound on to absorption carbon or finely divided coal. Compounds of the metals, chromium, tungsten, uranium, manganese, cobalt, nickel and iron are indicated as suitable and detailed yields are given for ferric hydroxide and Prussian blue. H. C. R.

1014. Valuable Products from Coal and the like Solid Carbonaceous Materials, Asphalts, Various Petroleum Products, Tars, Phenols, Cresols and the like by the Action of Hydrogen at Elevated Temperatures and Pressures. H. D. Elkington, Assr. N. V. de Bataafsche Petr. Mij. E. P. 348,243, 14.5.31. Appl. 20.2.30.

A scheme for the hydrogenation of coal, asphalt, phenolic and cresolic substances, etc., is outlined in which a catalyst consisting of a metallic sulphide is used. The preparation of the catalyst consists of contacting the metallic oxide or hydrated metallic oxide with sulphuretted hydrogen. H. C. R.

1015. Valuable Hydrocarbons from Coal, Tars, Mineral Oils and the Like. J. Y. Johnson, Assr I. G. Farbenind A.-G. E.P. 348,252, 14.5.31. Appl. 22.2.20.

To obtain the desired temperature in the hydrogenation process, oxygen-containing substances of carbon are added to the reactants. Reduction of these yields the heat required for the hydrogenation proper. H. C. R.

1016. Treating Coals, Tars, Mineral Oils and the Like by Hydrogenation under High Pressure and at High Temperature. J. E. Löffler. E.P. 348,449, 14.5.31. Appl. 3.7.30.

A plant for the hydrogenation of coal, oil and the like is outlined in which the heating and cooling of the reaction products and the reactants is performed by high pressure steam led in closed circuits throughout the plant. H. C. R.

1017. Catalysts for Use in the Hydrogenation of Carbonaceous Materials. I. G. Farbenind A.-G. E.P. 348,690, 15.5.31. Appl. 15.2.30.

The outlines are given for the preparation of catalysts containing sulphur, tellurin and selenium in which the metals of the second and sixth groups of the periodic system or their compounds are treated with the hydrides of the aforementioned three elements, or any other volatile compound of the same. H. C. R.

1018. Valuable Products from Coal, Tars, Oils and the like Carbonaceous Materials. H. D. Elkington, Assr. N. V. de Bataafsche Petr. Mij. E.P. 348,721, 22.5.31. Appl. 21.2.30.

Catalysts and their preparation are indicated for use in hydrogenation of carbonaceous materials. The oxides of iron, cobalt, nickel manganese, tungsten, molybdenum, uranium or the metals themselves serve as catalysts and can be reactivated by heat alone or with steam. H. C. R.

1019. Destructive Hydrogenation of High Boiling Hydrocarbons. Gas Light and Coke Co. E.P. 348,803, 21.5.31. Appl. 29.3.30.

The invention proposes to take advantage of the fact that the chemical composition of the products of hydrogenation depend largely on the composition of the original material. For example, a gas oil blend, which contains 68% paraffins, 4% unsaturated hydrocarbons, 19% aromatics, and 9% naphthenes, when subjected to hydrogenation in the presence of molybdenum and boron at a temperature of 460° C. and a pressure of 200 atm. produces a spirit containing 48% paraffins, 3% unsaturated compounds, 29% aromatics and 20% naphthenes.

By blending paraffin compounds with close chain compounds the reaction is more easily controllable otherwise the strongly endothermic reaction makes it difficult to control and maintain at the correct temperature. R. P.

1020. Method of Producing Liquid Hydrocarbons. J. Maruhn and L. Tübben. U.S. Pat. 1,807,122, 26.5.31. Appl. 10.3.30.

The invention consists in the use of a suitably designed hydraulic press for the hydrogenation of coal or oils. It is possible to use finely pulverized coal without any addition of oil. In the compression space a direct and intimate mixture of the materials is attained and hydrogenation can be carried out under high pressure and at a temperature of any height.

The press consists of a cast iron body holding a steel cylinder into which is inserted the working cylinder. A coil is cast in the steel cylinder, through which hot oil may be pumped to heat the apparatus. A plunger fits the cylinder and at the closed end of the cylinder a passage is provided with means for opening and closing. A tube passes through the side of the cylinder and case, through which the materials may be passed into the press. Details of operation of the press are given in D.R.P. 350619. R. P.

1021. Cracking, Catalysing, and Hydrogenation of Carbonaceous Materials. A. E. Bianchi and G. Guardabassi. U.S.P. 1,810,387, 16.6.31. Appl. 30.11.26. Conv. (U.K.) 27.5.28.

The invention relates to the cracking and hydrogenation of oils, tars or coals at atmospheric pressure. The apparatus consists of a retort having curved sides uniting in a longitudinal ridge at the centre of the retort bottom. A second retort is located under the ridge. Agitators are located on each side of the ridge in the upper retort and centrally of the lower retort. The material is fed into the upper retort at one end, travels through the retort and passes through an opening at the other end into the lower retort.

Above and in communication with the retort is a chamber containing a reticulated screen from which fine chains depend and through which the vapours and gases pass out of the apparatus. These chains may be coated with reduced nickel or other catalyst to promote hydrogenation of the cracked products. R. P.

Chemistry of Petroleum.

1022. Comptes Rendus de la Dixieme Conference Union Internationale de Chimie. Liège. 16.9–20.9, 1930. Pp. 1–72.

The report summarises the proceedings of various council meetings, the general assembly, etc., and includes brief reports of the work of the several committees. The question of unification of chemical abstracts was discussed and a committee representing Great Britain, Germany, France and U.S.A. is to be appointed to consider this problem.

To the petroleum chemist the report of the Committee on the Reform of the Nomenclature of Organic Chemistry should be of interest, as it includes a section on hydrocarbons (pp. 58–59). The recommendations closely follow current practice, the generic names of the various groups being as follows (subject to adaptation by the sub-committees to the various languages) :—

(*a*) Saturated open-chain hydrocarbons—alkanes.
(*b*) Unsaturated open-chain hydrocarbons with double bonds—alkenes, alkadienes, alkatrienes, etc.
(*c*) Unsaturated open-chain hydrocarbons with double and triple bonds—alkenynes, alkadienynes, etc.
(*d*) Unsaturated open-chain hydrocarbons with triple bonds—Alkynes, alkadynes, etc.
(*e*) Monocyclic saturated hydrocarbons—cycloalkanes, etc.

In the report of the Committee on Thermochemical Data, the use of benzoic acid as standard for the determination of heat of combustion is discussed, the value 6319 cals. 15° per gramme being adopted for this substance, the latter being weighed in vacuo. C. C.

1023. Thermal Decomposition of Organic Compounds from the Standpoint of Free Radicals. I. Saturated Hydrocarbons. F. O. Rice. *J.A.C.S.*, 1931, **53**, 1959–1972.

The demonstration of the existence of simple alkyl radicals by recent workers makes it possible to examine the mechanism of the decomposition of organic compounds from the standpoint of free radicals with some confidence. When a hydrocarbon decomposes, for example, under the influence of heat, it may be assumed that it dissociates into two free radicals which fly apart. A comparison of the strength of the C–H bond (93·3 kg. cal.) with that of the C–C bond (71·0 kg. cal.) shows that the dissociation occurs only through the rupture of a C–C bond and that any breaking of a C–H bond is wholly negligible compared with that of the C–C bond. The probability of the two radicals recombining is negligibly small, so that the free radicals formed can only decompose or react with surrounding molecules. Thus a free methyl group can take a hydrogen atom from a hydrocarbon molecule to give methane and a hydrocarbon radical. An ethyl radical, however, can react in one of two ways : (*a*) similarly to the methyl radical, to give ethane and a hydrocarbon radical, or (*b*) decompose to give ethylene and hydrogen. Higher radicals than ethyl can decompose similarly to give an ethylene hydrocarbon and a hydrogen atom or free radical. The dissociation of radicals is possible because in the process the single bond becomes a double bond, a process of internal compensation which releases 52,800 cal., this substantially contributing to the minimum activation energy required to break a C–H or a C–C bond. The decomposition of paraffin hydrocarbons can be represented as a chain type of reaction in which free hydrogen atoms or free radicals combine with one of the hydrogen atoms of the surrounding hydrocarbon molecules. The hydrocarbon radical resulting then decomposes into

a compound and either a smaller radical or a hydrogen atom. This process can, of course, be repeated a large number of times. The decomposition of the products is determined, therefore, almost exclusively by the chain cycle and is independent of the primary decomposition of the hydrocarbon. It is thus possible to calculate the composition of the products when a hydrocarbon is allowed to decompose to a small extent. In these calculations all primary C–H bonds are considered to have the same strength, all secondary C–H bonds 1200 cals. less and tertiary C–H bonds 4000 cals. less than the primary C–H bond. An arbitrary assumption necessary in the calculations is the assignment of the relative strengths of the different classes of C–H bonds. The decomposition of several simple paraffin hydrocarbons is discussed.

S. F. B.

1024. Heat Capacity and Free Energy of Formation of Ethane Gas. V. R. Thayer and G. Stegeman. *J. Physical Chem.*, 1931, **35**, 1505–1511.

The heat capacity of ethane gas has been measured at various temperatures by means of a constant flow calorimeter similar in design to that of Scheel and Heuse (*Ann. Physik.*, 1912, **37** (4), 79). The values obtained, together with those of other investigators, are shown graphically. Utilising the specific heat data obtained, the free energy of ethane gas has been recalculated, the equation obtained being—

$$\Delta F = -18{,}789 + 16{\cdot}17\ T \ln T - 0{\cdot}006\ T^2 - 63{\cdot}9\ T.$$

W. D. S.

1025. Application of Nickel Catalysts in the Synthesis of Benzene. F. Fischer and K. Meyer. *Brennstoff Chem.*, 1931, **12**, 225–232.

Nickel catalysts, when produced under special conditions, can hydrogenate carbon monoxide under normal pressures to form methane and liquid hydrocarbons. The reaction can also be directed so that the formation of methane is further repressed and about two-thirds of the carbon monoxide consumed are converted into liquid products.

The nickel is decomposed in the form of the carbonate in the presence of an activator. An addition of 18% of thorium oxide and a quantity of kieselguhr in the proportion of 1 : 1 to the quantity of metal used are found to be the best activators; potassium carbonate is utilised as precipitant. The nickel catalyst is then reduced with hydrogen at a temperature of 450°, although this temperature can be lowered if some copper is added.

The original gas utilised in this research was obtained from a mixture of water-gas and cracked gas and contained 26–27% CO, 55–57% H_2, 3–4% CO_2, 6–7% CH_4 and 7–8% N_2.

Of the total consumption of carbon monoxide, 65% was converted into liquid and 35% into gaseous hydrocarbons. The catalyst was found to be very stable and if desired thorium can be replaced by manganese.

W. S. E. C.

1026. Physical Constants of Petroleum Fractions. W. L. Nelson, *Petr. Eng.*, 1931, **2** (8), 41.

References are given to a number of articles giving data on the physical constants of petroleum hydrocarbons and fractions. The molecular weight determined by the cryoscopic method of a petroleum fraction of boiling range below 900° F. is generally similar to that of a normal paraffin of the same boiling range; above 900° F. the normal paraffins have higher molecular weights. A curve showing the relation between molecular weight and boiling point of paraffin hydrocarbons and average petroleum oils is given. Refining treatment of oils is stated to lower the molecular weight considerably.

O. G. Wilson's nomographic chart for the correction of boiling point from one pressure to another, is reproduced.

Reference is made to the Nat. Gas. Assocn. report on the vapour pressures of paraffin hydrocarbons from heptane to pentadecane.

Boiling point corrections of average petroleum fractions from vacuum to atmospheric pressure are shown to be greater than those of normal paraffins. Acetylenes give smaller corrections, whereas naphthenes, olefins, iso-paraffins and aromatics give higher corrections than normal paraffins. C. L. G.

Analysis and Testing.

1027. Fractional Distillation of Crude Oil. E. O. Slater. *Oil Bull.*, 1931, **17**, 272–4.

The four-point distillation method for evaluating crude oils is described in detail. It consists of a preliminary distillation (topping to a vapour temperature of 500°) under specified conditions, of a weighed sample of the crude oil. Above 250° F. the distillation rate is not to exceed 10 ml./min. A 300 ml. sample of the tops (containing the gasoline and kerosine) from this distillation is rerun in the A.S.T.M. apparatus (D–285–30 T). The distillation rate is 2–3 ml./min. for the first 5 ml. distilled and 4–5 ml. thereafter. The volumes of distillate in ml. are determined when the vapour temperature reaches the following points: 221° F., 284° F., 392° F. and 437° F. From these volumes the percentage of gasoline recovered at each of these temperatures and the percentage of gasoline in the crude oil are calculated. The percentage of tops is multiplied by the percentage of net tops calculated from the crude distillation (from the net volume of crude oil and the net tops or total tops minus water), and the resulting figure is multiplied by a different factor for each temperature 5, 2, 1·11 and 1, respectively, for the four distillation points to give the percentage of gasoline in the crude oil. W. S. E. C.

1028. Water Content of Fuels and its Determination. M. Kremer. *Petr. Zeit.*, 1931, **27** (24), 443–5.

In the earlier work on this subject Dixon, Hubendick and Wawrzinick determined the cloud-point of mixtures of alcohol and hexane or benzol at different temperatures. Water was added to these mixtures until they became turbid and the temperatures noted at which turbidity vanished. Another method made use of magnesium amalgam. This was allowed to react on the mixture of alcohol and hydrocarbons in a specially constructed apparatus and the hydrogen evolved measured. Dixon's values show that benzol-alcohol mixtures compared with hexane-alcohol mixtures are insensitive to a dilution with water at high temperatures, whilst on the other hand hexane-alcohol mixtures are more insensitive to dilution with water at lower temperatures. The sensitivity towards water is greater for both mixtures, the less the content of alcohol in the mixture.

Heale's method is next described. It can be applied to determined quantities of water as small as 0·05% and has been tested for alcohol-hydrocarbon mixtures. The method is based on the formation of anhydrous conversion products—*e.g.*, $Al_2(OC_2H_5)_4O$ and $Al_4(OC_2H_5)_6O_3$ obtained from normal

aluminium ethylate. These compounds, when dissolved in organic solvents such as xylol, give a voluminous precipitate of aluminium hydroxide when very small traces of water are present in the fuel. The compound $Al_4(OC_2H_5)_6O_3$ is most sensitive to humidity. In practice 50 c.c. of the fuel mixture are taken and 2 c.c. of the reagent (dissolved in xylol) added. The mixture is shaken, left to stand for from 2 to 3 hours, when the $Al(OH)_3$ is deposited in a thick layer of a definite height from the bottom of the vessel according to the water content of the fuel. This method is so far only qualitative, but the suggestion of filtering the hydroxide, burning and weighing the aluminium oxide formed is proposed in order to obtain quantitative results.

W. S. E. C.

1029. Practical Analysis of Lubricating Greases. Dr. W. T. Sieber. *Petr. Eng.*, 1931, **2** (9), 96.

The method recommended by the A.S.T.M. is desirable for very accurate work, but requires special care, is complicated and requires too much time for practical control work in a grease-making plant. A general outline of the methods suggested for control work are: (1) Physical tests. (*a*) Consistency. This is determined by A.S.T.M. penetrometer on a worked sample of the grease if the penetration is not greater than 350 mm./10 at 77° F. For softer lubricants the MacMichael torsion viscometer is used, and determinations also made at 77° F. (*b*) Melting point. The bulb of an accurate thermometer is coated with grease and suspended in an empty test tube which is placed in an oil bath. The bath is heated at the rate of 5° F. per min. and the temperature at which a drop of grease falls to the bottom of the tube is recorded as the melting point. (*c*) Colour, odour, flash and fire points are determined by the usual methods. (2) Chemical tests. 200/400 gr. of grease are dehydrated by heating to 105° C. for about one hour and introduced into a weighed aluminium cup which fits into a centrifuge tube. Petroleum ether is added and the grease broken up with a glass rod. The mixture is centrifuged for 12/15 min., the petroleum ether solution decanted off, and the process repeated three or four times, until the petroleum ether is no longer discoloured. The petroleum ether used should be neutral in reaction, leave no oily residue on evaporation and have an E.P. not higher than 210° F.

The extracts are mixed and introduced into a weighed distillation flask, the petroleum ether distilled off, and the weight of the remaining material determined. Aliquot portions are examined in order to determine the percentage of free fatty acids, traces of soap fatty acids and the free fats which may be present. The percentage of mineral oil is obtained by difference. The extracts are used to ascertain the character of the mineral oil and fatty matter present in the grease. The solid matter remaining in the aluminium cup consists of the filler and the bulk of the soap present and is examined accordingly. The percentage of water present in the grease is determined by the A.S.T.M. method. A schematic diagram of the steps taken in the chemical examination is given.

R. A. E.

1030. Determination of the Benzene and the Normal Hexane Content in a Mid-Continent Petroleum. J. H. Brunn and M. M. Hicks-Brunn. *Bur. Stan. J. Res.*, 1931, **6**, 870.

The benzene content of a crude oil from Kay County, Okla., was determined by repeated fractionation of the lighter fractions, taking off in the final distillation 37 one degree cuts between 44° C. and 81° C. Each fraction was nitrated, using a large excess of acid and agitating vigorously for a long time at below 10° C., owing to the rather low reactivity of benzene towards

nitrating acid. Recrystallisation of the nitrated material gave pure m-dinitrobenzene of melting point 89·7° C. In the presence of hexane a constant boiling mixture is obtained. Practically all the benzene was found in the fractions boiling between 65° C. and 75° C., the peak being in the fraction boiling between 69° and 70° C. The total quantity present in the crude oil was of the order of 0·08%.

Pure benzene could also be removed by extraction with aniline, followed by washing with HCl and water and by redistillation.

N-hexane was isolated by agitating the fraction boiling at about 69° C. with chlorosulphonic acid, separating the sludge, washing the hydrocarbon layer with NaOH solution and drying over $CaCl_2$. The hexane was then purified by redistillation over sodium. About 0·3% of normal hexane was found present in the crude oil, the isomers not being present to nearly such an extent. All the fractions boiling between 67° and 72° C. consist mainly of n-hexane, the minor constituents being probably 3—methyl pentane, benzene and methyl cyclopentane.

The physical properties of the extracted benzene and hexane were determined, these figures agreeing well with those of the synthetic products.

A full description of the continuous b. pt. apparatus placed between the top of the column and condenser for the purpose of determining the true boiling points is given, together with data on the yields from the different fractions and physical constants of the purified hydrocarbons. C. L. G.

1031. Testing the Purity of Solid or Liquid Hydrocarbons. A. Esau. E.P. 345, 464, 26.3.31. Appl., 5.3.30. Conv. (Ger.), 13.3.29.

A method is proposed in which the material to be tested is subjected to the action of short electric waves (10^8 Hertz) and the resultant changes of temperature therein are measured. From the data obtained the purity of the material in terms of some definite standard may be determined. The material is tested either in the electric field within a condenser or in the magnetic field within a coil, a thermo-couple being employed for measuring the resultant changes of temperature in the material. A particular application of the invention is the determination of the water content of transformer oils.

W. H. T.

See also Abstract No. 978.

Anti-Detonation.

1032. Gasoline Dopes. H. C. Dickinson. *Ind. Eng. Chem.*, 1931, **23**, 517.

"Dopes" are defined by the author as substances which, when added to petrol in amounts not exceeding about 1% by weight, improve appreciably some property or properties of the petrol as an engine fuel. The following properties are dealt with:—

Starting Properties.—In view of the rich mixture supplied for starting by a well-designed modern carburetter and the small proportion of fuel that therefore needs be vaporized to give an inflammable mixture, it is not considered probable that starting can be greatly improved by any material added to the fuel in minute quantities.

General Volatility.—The general volatility of a fuel, which affects acceleration, dilution of lubricating oil, etc., is indicated satisfactorily by the 90%

point on the A.S.T.M. distillation curve. No substance has been suggested as a dope to affect this value appreciably as the chemical nature of the fuel would have to be radically altered.

Pinking.—Certain dopes are well known to reduce pinking when added to petrols in minute amounts, but in spite of intensive research no fresh anti-knock dopes have appeared for some time, and fresh claims should be treated with reserve until full tests have been made.

Power Output.—For a given carburetter setting, increased B.M.E.P. can only be obtained either by increasing the latent heat of the fuel—and, hence, the charge density—or the calorific value of the mixture. It is improbable that any dope could affect these fuel characteristics to a marked extent.

Carbon Deposits.—Certain solvents are known which will remove the binder from the deposits in an engine and enable the carbon to be blown out of the exhaust. These do not come under the category of dopes. Often hours of running at high load under favourable conditions will probably reduce the deposits more appreciably than any dope advertised.

Fuel Consumption.—Since many carburetters are set over-rich to get easy starting, a dope that will induce or compel drivers to run on a leaner mixture may improve consumption and performance. There are certain dopes which may affect the rate of fuel feed through the jets and so produce an improvement. Carburetter adjustment would have accomplished the same or better results. G. B. M.

1033. Detonation in Internal Combustion Engines. K. Schnauffer. *Z. ver. Deut. Ing.*, 1931, **75**, 455-6.

Schnauffer utilises in this research a single-cylinder engine in which the firing spark plug is located at one end of the cylinder head. Three other spark plugs are located along the direction of flame travel. These are connected to a source of high-frequency current and to an oscillograph. The passage of the flame front ionises the gases between the electrodes, thus allowing the passage of the current and giving a record on the oscillograph. The firing is also recorded, as well as the pressure-time curve, and the oscillations of a timing fork. In the absence of detonation, the flame travels at an increasing speed, covering the 13 cm. of travel in 0·005 to 0·01 sec. When the engine is made to detonate, the flame reaches the last two indicators practically simultaneously. This shows that the detonation consists in the spontaneous ignition of the whole mass of unburned gas compressed in front of the flame. W. S. E. C.

1034. "Octane Number" and What It Means as Applied to Motor Gasoline. A. L. Foster. *Nat. Petr. News*, 1931, **23** (24), 30–32.

In January, 1931, the C.F.R. Steering Committee adopted the use of a mixture of normal heptane with iso-octane as a standard reference fuel for testing knock in gasoline.

The octane number is the percentage of iso-octane in normal heptane, the heavily knocking component, required to give the same degree of knocking as the fuel being tested, under a definite set of engine conditions.

As heptane-octane fuel is very expensive, two secondary reference gasolines have been prepared by the Standard Oil Co. of New Jersey. These secondary fuels will be used all over the country. The fuels are standardized against the primary fuel, the heptane-iso-octane mixture, and will be matched with the gasolines tested by the addition of differing amounts of lead tetra-ethyl. The corresponding knock ratings will be found from curves prepared for the purpose. P. A.

1035. NEED FOR STANDARDIZING KNOCK TESTS. P. F. Swanson. *Oil and Gas J.*, 1931, **30** (5), 46.

The present methods of determining the chemical composition of gasolines are known to be unsatisfactory and are incapable of providing a measure of the anti-knock value of a fuel not only on this account but also because of the variation in anti-knock value of the individual members of each class of hydrocarbon. An endeavour has been made to discover a more satisfactory method of laboratory examination, starting with five pure aromatic hydrocarbons and one pure hydrocarbon from each of the naphthene, iso-paraffin and unsaturated groups. Each hydrocarbon was blended with an equal quantity of Mid-Continent paraffin-base gasoline. Sulphonating the blends with successive acid washes of increasing strength was found to be an unreliable method, since it does not give an exact group separation. B. pt./grav. relationship was next tried. Charts are presented which show this relationship for pure hydrocarbons of the four groups and also for 2½% fractions of three gasolines of widely different type. It is shown that the method gives an interesting indication but cannot be used as a precise method for routine testing and comparison of anti-knock value. In the absence of a satisfactory chemical test the only reliable method is to determine the performance of the fuel in the engine itself, so arrangements were made to examine 18 samples of gasoline of widely different character in the test engines normally employed in five different laboratories. These were: Texas Company, Horning (fixed compression type); Michigan University, N.A.C.A. (variable compression); Sinclair Refining Co., Waukesha (fixed compression); Kansas City Testing Lab., Novo (variable compression); and Continental Oil Co., Ethyl S. 30.

A full description of each engine and of the method of operation is given. The composition of the samples is stated and the results obtained by the five laboratories are tabulated and graphed. The results show that the Texas Co. and Michigan University benzole values and octane numbers are in fair agreement, but the Kansas City Testing Lab. benzole equivalents are based on an entirely different scale. The Continental Oil Co. results show that the number of c.c. of T.E.L. which must be added to gasolines having a similar octane number as determined by Michigan University, in order to equal Ethyl standard quality, varies considerably according to the origin of the gasoline. For three gasolines of octane number 55, the range was 1·4/3 c.c. of T.E.L. Consequently ethylization is not considered a satisfactory method for comparing anti-knock values. The M. W. Kellogg Co. therefore operates its S. 30 engine by matching the fuel under test with blends of pure benzene and Penna straight-run gasoline of E.P. 400° F.

For estimating values of blends measurement of increases in compression ratio is the best criterion, the anti-knock value of 50/50 blends closely approximating the mean value of the constituents. It is believed that results of sufficiently comparable value for commercial purposes can be obtained with any type of engine provided the routine method of test is strictly standardized and calibrated against iso-octane/heptane blends. The standard engine advocated by the C.F.R. will only be essential when results are required for general interpretation. There is need for further investigation of the relation between anti-knock value and the chemical and physical structure of gasolines leading to research into methods for producing gasolines of higher anti-knock value.

R. A. E.

Engines.

1036. JALBERT HEAVY OIL ENGINE. W. F. Bradley. *Mech. Eng.*, 1931, **53**, 452. *Automotive Industries*, 1931, **64**, 548–549.

This engine belongs to the class using a carburettor and a premixing and compression cylinder.

The engine is a six-in-line, bore 4·92 in., stroke 7·09 in. and expected to develop 180 H.P. at 1930 r.p.m.

In place of the fuel pump and injector of the Diesel engine, the Jalbert has a carburettor which feeds a small cylinder mounted in the head of each working cylinder.

The engine is said to be similar in principle to one known as Romeiser, described in *New York Times*, March, 1931. W. A. W.

1037. DEVELOPMENT OF THE HIGH-SPEED OIL ENGINE IN ENGLAND. A. E. L. Chorlton. *Mech. Eng.*, 1931, **53**, 421–426.

An interesting survey is given including diagrammatic sketches, commencing with the original oil engine patents of Akroyd Stuart in 1890 and concluding with the Diesel type engine as constructed for R.101.

Data is quoted of tests carried out with the early types of Diesel engines. W. A. W.

1038. DOUBLE-ACTING SULZER MARINE DIESEL ENGINE. *Mar. Eng.*, 1931, **54**, 179.

A description is given of the first large Sulzer double-acting two-stroke marine oil engine at present undergoing test; normal rating 7600 H.P., r.p.m. 106, 8 cyl. each measuring 700 mm. by 1200 mm.

Scavenging is effected at the top and bottom of the cylinder through superimposed double rows of ports. No troublesome eddies are created during scavenging of the lower end of the cylinder through which the piston rod passes. The special cast iron piston and piston rod are cooled with water which enters the piston rod below the crosshead. Adjustment of the compression space is facilitated by the piston rod being secured by a nut above and below the crosshead. The fuel pump plungers, which are of the packing-less type, are driven from the crossheads of the compressors. All the fuel valves are so designed that they can be dismantled without removing the operating levers. C. I. K.

1039. WASTE-HEAT BOILERS IN DIESEL ENGINES. *Mech. Eng.*, 1931, **53**, 452–453. *Marine Eng. and Shipping Age*, 1931, **36**, 183–186, 192.

There are two general services to which steam generated in an exhaust-gas boiler can be applied: (1) Operation of auxiliary machinery; (2) heating the crew's quarters, passengers' accommodation and taking care of galley requirements.

The capacity of the waste-heat boiler is governed primarily by the power and type of Diesel engine to which it is connected, and secondly by its own design.

Progress in design and construction of waste heat boilers has kept ahead of the demand and regarding this development reference is made to Major W. Gregson's paper presented to D.E.U.A. on Dec. 12th, 1930, wherein Gregson stated that a tanker powered with a four-cycle engine of 2500 b.h.p. will generate 2500 lb. of steam per hour at 120 lb. pressure, also on a large motorship of 20,000 b.h.p., equipped with four-cycle engines, he estimated waste-heat boiler steam output of 20,000 lb. per hour at 120 lb. per square inch.

Reference is made, giving details, of a waste-heat boiler which is manufactured by the Foster Wheeler Corporation. W. A. W.

1040. Mechanism of the Atomization of Liquids. R. A. Castleman. *Bur. Stand. J. Res.*, 1931, **6** (3), 369.

Atomization of liquids as far as concerns the internal combustion engine may be of two types: (1) air stream atomization where air at high speed is passed over the surface of the liquid, as in carburettor engines and (2) solid or airless injection where the liquid is forced under very high pressure into still air, as in high-speed Diesel engines. The theory is developed that atomization takes place firstly through the tearing off of ligaments from the main mass of the liquid by the air stream, this being followed by collapse of the ligaments into discrete droplets. Rayleigh's work (*Proc. Lond. Math. Soc.*, 1879, **10**, 41) on the rate of collapse of liquid columns is assumed to apply to such ligaments. The mechanism of the collapse of these ligaments is illustrated by reproductions of Scheubels spark photographs of the atomization of water and alcohol in an air stream of varying speeds through a model of a carburettor throat. The ligaments torn off at low air speeds are clearly seen, while at high speeds these appear much finer and at 10,500 cm./sec. vanish, the small drops appearing to be directly torn from the large mass. Reference is made to Sauter's photometric measurements of the mean size of the drops formed when water is atomised in an air stream. In a high-speed air stream 10/12,000 cm./sec. the radius of the particle is taken as $5\ \mu$. At this speed true atomization can be said to occur, since this is the minimum size of drops formed, agreeing in the main with Scheubel photographs of atomization at this speed.

From a consideration of Rayleigh's work on the rate of collapse of liquid columns and Sauter's measurements of particle size, the values of the length/diameter ratio of the ligaments and their degree of instability is calculated. Determination of the size and instability of the ligaments from the size of the drops of the spray shows that at high speeds the ligaments collapse so quickly, *i.e.*, when true atomization sets in, that the droplets appear to be picked up from the main mass as has been observed photographically. C. L. G.

1041. Separately-Scavenged Two-Cycle Airless-Injection Engine. *Engineering*, 1931, **131**, 120.

A detailed description of the "Sandon" Diesel valveless oil engine is given, with curves of output, efficiencies and fuel consumption. The engine is of the airless-injection two-stroke type and differs from the usual forms in that the scavenge air, supplied by a valveless rotary air compressor driven by gearing from the engine crankshaft, is independent of the crankcase. This allows forced feed lubrication of the working parts. Two oil systems are provided so that different oil can be used for the pistons and the bearings. The unused oil is caught in trays and after treatment is returned to the system. The air compressor also supplies ventilating and cooling air to the crankcase and can be used to charge a receiver for starting the engine from cold. J. M. F.

1042. Fuel Injection Device for Internal Combustion Engines. S. A. Larsen. E.P. 343,378, 19.2.31. Appl., 9.4.30.

The invention relates to improvements in devices for fuel injection in engines using airless injection and in which a pressure accumulator in the pressure-pipe system of the fuel pump is used to effect injection.

The accumulator is charged by a fuel pump and injects the fuel to the engine when the pressure in the pressure-pipe system is released. The differential or step piston of the accumulator, either spring or fluid pressure loaded, is exposed to a smaller pressure than the injection pressure during charging, and by a valve arrangement maintains communication between the two pressure

chambers of the accumulator. When the pressure in the pressure-pipe system is released this valve operates and isolates the charge of fuel to be injected into the engine. This fuel is acted upon by the higher pressure piston of the differential piston and injected under the full injection pressure. The pressure in the pressure-pipe system is released by an overflow slide valve, which determines the commencement of the effective part of the delivery stroke of the fuel pump, and regulates the amount of fuel injected. J. M. F.

1043. Carburettor for Internal Combustion Engine. E. Pohjanpalo. E.P. 344,958, 19.3.31. Appl., 19.12.29.

The invention describes a carburettor that can be used for fuels ranging from petrol to crude oil. This is effected by providing a heating chamber between the float chamber and an orifice controlled by a needle valve. This valve is enclosed in fine meshed gauze which partly atomises the liquid fuel drawn through the orifice and aids in the mixing of the fuel and the air, the latter being drawn through an automatic regulator. A flap is placed between the heating and the float chambers so that in the event of an explosion this does not spread to the float chamber. The hot mixture flows past guide wings which impart a whirling motion to it for better mixing, and is cooled by drawing in supplementary air. The temperature in the heating chamber is controlled by a valve in the exhaust passage passing round the chamber, to suit the fuel being used. J. M. F.

See also Abstract No. 972.

Coal and Shale.

1044. Italian Shale Oil Development. *Chem. Trade J.*, 1931, **88**, 488.

The U.S. commercial attaché at Rome states that an agreement has been signed between the Italian Government and an Italian Company known as A.B.C.D. subsidising the production of crude oil from shale. Deposits of bituminous limestone near Ragusa and Vizzino in Sicily are to be worked up to produce 50,000 tons oil yearly, the yield from the shale being given as 12%. The subsidy amounts to 140 lire per ton of oil of specified grade. C. L. G.

See also Abstracts Nos. 1013–1018, 1020, 1021.

ABSTRACTS.

Geology.

1045. Natural Gas from Oriskany Formation in Central New York and Northern Pennsylvania. P. D. Torrey. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 671–686.

Much leasing and drilling activity has resulted in central New York and northern Pennsylvania by the recent discovery of two large gas producing fields from the Oriskany Sandstone, which is the basal bed of an almost complete series of Devonian rocks. This consists of black carbonaceous and bituminous shales with a few interbedded limestones.

The Tyrone field, from which production has been very satisfactory, was considered very favourably before the discovery well was drilled; several wells previously drilled in the small east syncline encountered water-bearing Oriskany Sandstone.

The Oriskany Sandstone is believed to be the producing horizon of both the Tyrone and Farmington fields.

The Farmington field—though by no means defined at present—is located on the Sabinsville Anticline, and the discovery well is reported to have had an initial open flow of 12,000,000 c. ft. Three other producing wells have since been completed.

Owing to irregular conditions of sedimentation the occurrence of the Oriskany both at outcrop and as proved by drilling is extremely variable; as developed in central and western New York it probably represents submarine bars. Careful study of its sedimentation is said to be as essential to the successful location of new gas fields as the mapping of the anticlinal structures.

Structure contour maps and tables of well records are given in the paper.

G. S. S.

1046. Truncation of Maricopa Sandstone Members, Maricopa Flat, Kern County, California. E. R. Atwill. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 689–696.

It has been postulated in the past that no regularity of beds occurred in the older formation and that production has been due to the penetration of local sand pockets occurring 400–600 ft. below the Etchegoin-Maricopa contact; this is called the "Signal" zone. Subsequently saturated sands were proved at and immediately below the junction, and the new idea of a "contact" zone was accepted, though it was not understood why it was found in some wells while in other cases the beds below the contact were entirely replaced by shales.

The author attempts to solve the problem by means of a series of well sections and structural contour maps. An angular unconformity ranging from 5°–10° is proved between the Etchegoin and Maricopa formations, and it is shown that the sand members of the latter are truncated against the more gently dipping Etchegoin.

Evidence of an eastward plunging anticlinal nose is also proved in the Maricopa. It is believed this guides the accumulation in the "Signal" zone, and also localises in commercial quantities the oil in the truncated sandstone along its flanks and down the axis.

The sands appear thicker on the south flank of the nose. Here the largest producers are obtained, an initial production of from 3,000–4,000 brls. per day having been obtained as compared with 1,000–2,000 brls. on the north flank. G. S. S.

1047. Microscopic Subsurface Work in Oil Fields of the United States. R. D. Reed. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 731–754.

This paper deals with the present position of the application of the microscope to subsurface work in various oil fields of the United States. These include Kansas, West Texas, Rocky Mountain District, California, and Gulf Coast Tertiary and Cretaceous districts.

From information furnished the author by various workers, full discussion is given to the subject and comparisons are made with each area. The methods employed vary according to the rock-types penetrated, samples available, and to the nature of the problems involved. For example, micropalæontology is of most importance in areas of Mesozoic and Cenozoic rocks, micro-lithology in Palæozoic rocks and the Cretaceous of the Rocky Mountains.

Much success has been attained by the new methods, and further results of economic importance are considered likely in the future. A bibliography concludes the paper. G. S. S.

1048. Factors Governing Accumulation of Oil and Gas in Mirando and Pettus Districts, Gulf Coastal Texas, and Their Application to Other Areas. O. L. Brace. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 755–791.

By the construction of contour maps and cross-sections, the author has been able to show the structural conditions and lithology of the Mirando oil and gas area.

All the surface rocks are Tertiary in age and may be referred to the Claiborne formations or younger, the Wilcox being the last exposed farther west and met only in the deepest wells. Throughout the region the Pliocene Reynosa covers the Oakville and is in turn overlapped coastward by newer deposits.

Accumulation has occurred in narrow strike lenses produced under shore line conditions and in which lateral gradations in porosity has led to trapping. Present indications suggest that source material from which oil and gas are derived was originally deposited within or near to existing producing horizons. There is no evidence at the surface or below of any major movements; the coastal plain here being a gentle monocline.

The author considers that in the Pettus area the same factors control the accumulation of oil as in the Mirando district. The area of production lies within the same stratigraphic limits in both districts, and it is therefore concluded that the sands within the Frio-Yegua group constitute a potential source of oil and gas throughout the inner margin of the Gulf Coastal Plain of Texas. In the absence of structural influence it is thought the most effective means in the quest for new fields will be in following established strike trends. G. S. S.

1049. Taylor Age of San Miguel Formation of Maverick County, Texas. L. W. Stephenson. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 793–800.

Much confusion is said to exist in assigning the true age of the San Miguel formation. In this paper therefore the author reviews the evidence by re-examining available collections together with others from two additional localities in the field.

The formation has a thickness of about 400 ft. and consists mainly of calcareous sandstones and limestones. It contains a fairly long fauna, many species of which have a long range, and occur in both the Taylor Marl and the Navarro formation. Some of the species, however, are of more restricted age and indicate a Taylor age to the rocks. The San Miguel formation, further, contains a varietal form of *Exogyra costata* Say, but is lacking in the true *Costata* and other diagnostic Navarro species. The evidence indicates that an important faunal hiatus exists between the San Miguel and the base of the Escondido, a break which is partly filled by the non-marine Olmos formation.

The author concludes that the formation under review is Upper Taylor in age and not Navarro of Central Texas with which it has been generally correlated. G. S. S.

1050. Pre-Cretaceous Rocks Found in Wells in Gulf Coastal Plain South of Ouachita Mountains. H. D. Miser and E. H. Sellards. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 801–818.

In the part of the Gulf Coastal Plain south of the Ouachita Mountains, a large number of deep wells have passed through Paleozoic rocks which form a relatively smooth floor below gently inclined Cretaceous deposits. This floor has a steep southerly dip varying from 70 to 250 ft. to the mile, and consequently has not been reached by wells more than 25 miles S. or 40 miles S.E. of the Ouachita Mountains. In areas farther S.W., however, it has been penetrated in a trending belt extending from Oklahoma into Texas.

Wells are described from S.W. Arkansas, S.E. Oklahoma and N.E. Texas. The Paleozoic rocks reached in them are similar in character and age to those exposed in the Ouachita Mountains. Consisting mainly of shales and sandstones with an aggregate thickness of 25,000 ft., they range in age from Cambrian to early Pennsylvanian. Pre-Cambrian rocks have nowhere been reached.

The axis of the Ouachita geosyncline—that in which the rocks of the Ouachita Mountains were deposited—has an E.W. trend in Arkansas, but swings S.W. in Oklahoma and crosses Red River into Texas. The part of the geosyncline occurring in these States is markedly arcuate in form.

Detailed accounts of the wells follow with a table and map showing their location. G. S. S.

1051. Rocks Underlying Cretaceous in Balcones Fault Zone of Central Texas. E. H. Sellards. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 819–827.

Evidence obtained by well cores and cuttings in the Balcones Fault Zone has proved the existence of Paleozoic rocks underlying Cretaceous. In character and occurrence they are similar to the rocks of the Ouachita Mountains, consisting chiefly of shales and sandstones much folded and faulted. Graptolites were found and identified in black shale in the Bacon Well of Bell County at a depth of 1100 ft.

Deposition of the sediments is believed to have taken place in a trough or syncline in front of the land mass Llanoria. A map and well data is furnished of thirteen counties in and near the Balcones Zone where the rocks have been determined. G. S. S.

1052. Geothermal Variations in Coalinga Area, Fresno County, Calif. A. J. Carlson. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 829–836.

Data obtained from over 50 wells drilled in the Coalinga area are given showing variations of isothermal elevations in relation to structure.

The prevailing surface rocks are Tertiary and Cretaceous which dip into the valley and crop out successively in the area. Vertical sections through

the Eastfield anticline are also furnished illustrating the comparative trend of isotherms, underground strata, and ground surface.

The results indicate that the relative variations of temperature with depth are controlled chiefly by surface topography and increasing thickness of deposits. Further, occurrence of oil in the sediments does not affect to any extent the trend of rock temperatures. G. S. S.

Field Technology.

1053. Field Methods of Treating Oil in North Texas—South Oklahoma. C. R. Webb. *Oil & Gas J.*, 1931, **30** (6), 48.

In one of the fields in Texas production has been handicapped by clogging of the tubing with an unusual type of deposit, which occurred near the top of the flow string and extended downwards 200/300 ft. This deposit was found to consist mainly of silica, iron oxide, calcium and sodium chlorides, together with 7·79% of a waxy material soluble in petroleum ether. The deposit had to be removed either by the use of a paraffin cutter or by removing the tubing and melting the wax. In other fields difficulty has been experienced owing to the high B.S. & W. content of the oil. This has been overcome by a combined chemical and heat treatment which is described. Other wells have given trouble on account of the emulsifying tendency of the oil produced, and this has been overcome by injection of a water solvent chemical.

R. A. E.

1054. Pipe Line Currents and Soil Resistivity as Indicators of Local Corrosive Soil Areas. F. R. Shepard, *Bur. Stand. J. Res.*, 1931, **6**, 683.

The present tendency in the protection of pipe-lines against soil corrosion is to apply coatings in local areas selected for their resistance to the soil in that area, rather than to use a uniform coating over the whole line.

In many types of soil protective coatings are not necessary, while in others, which are usually areas of high soluble salt content either natural or due to contamination by waste products, etc., they are highly necessary. It is therefore very important to have a knowledge of soil conditions and to be able to locate corrosive areas.

The present investigation, carried out on nine pipe-lines ranging from the Gulf Coast to South Kansas, describes a method of locating these danger spots. The work of previous investigators indicated the presence of galvanic currents of varying densities over the length of the pipe-lines. These currents are shown to accumulate on the pipe-line for long distances and then discharge abruptly over relatively short sections. Measurements of the electrical resistivity of the surrounding soil showed that these areas of abrupt discharge were always associated with soils of low resistivity, while the areas of collection or little discharge were in soils of high or normal resistivity. In the areas of low resistivity and rapid discharge the pipe-lines were invariably severely pitted and rusted. An investigation of the origin of the galvanic currents suggested that they were due to: (1) Potential difference between bright iron and scale; (2) P.D. in different parts of the pipe due to strains, inequalities or impurities; (3) P.D. between different types of pipe or between new and old pipe; or (4) P.D. resulting from soil differences. It was established that the P.D. in the case of (2) was of the order of millivolts only, while in (4) it was of the order of 0·1 volt. Generally, galvanic action is attributed

to local action rather than to soil differences. This is borne out by the fact that currents are always found entering tank farms rather than leaving them, which would not be expected if they were due to soil differences. Such currents would be due to a difference of potential between the pipe-line and the tank bottoms. It was estimated that if the resistivity of the soil surrounding any particular length of line was less than 500 ohms. severe corrosion took place, while if above 1000 ohms. there appeared to be no connection between corrosion and resistivity. Full details of the methods of determining the density of the pipe-lines currents and the resistivity of the soil are given with the actual results obtained on the pipe-lines examined. C. L. G.

Crude Oil.

1055. Paraffin Crude Oils for Anti Knock. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **30** (7), 16.

Crude oils from the Michigan fields yield straight-run gasolines which are almost entirely paraffinic in character and consequently possess a very low anti-knock value. Analyses of nine crude oils from various fields in Michigan, and the yields of products which can be obtained from them, are given in the form of tables. Crude having an A.P.I. grav. of 41·2° from Mount Pleasant Pool, has been further examined, and the yields and properties of six straight-run gasolines varying in E.P. from 298° F./435° F. are given; 14·4% of a gasoline having an E.P. of 298° F. and possessing an octane number of 52, determined in an ethyl S. 30 engine, and 40·7% of a gasoline having an E.P. of 435° F. and an octane number of 15, are the limiting values determined. Topped residue (A.P.I. grav. 34·5°) from the crude, on cracking by the flashed residuum method at pressures of 250/300 lb. per sq. in. and at a temperature of 945° F., provided: (1) 62·3/63·6% of cracked gasolines ranging in I.B.P. between 86 and 94° F., in E.P. between 388 and 404° F., and in octane number between 67 and 68; (2) 28·0/33·8% of flashed residuum ranging in A.P.I. grav. between 10·0° and 12·3°, in B.S. and W. content between 0·3 and 0·6%, in visc. Furol at 122° F. between 20 and 36 secs., and possessing in all cases a cold test below 0° F.; (3) 3·9/12·2% of gas, coke and loss.

Mount Pleasant crude is somewhat easier to refine than that from Muskegon, and both the straight-run and cracked gasolines from the latter crude are lower in knock rating than corresponding products from Mount Pleasant crude. Muskegon gasoline of E.P. 437° F. required the addition of 37% of benzol to equal ordinary Pennsylvanian straight-run gasoline in anti-knock value, and Muskegon gasoline of 375° E.P. required the addition of 18% of benzol.

The properties of the benzines, kerosines, gas oils and lubricating oil distillates obtained from the nine crude oils are summarised and discussed.

R. A. E.

1056. Refining Crude Oil from Venezuela. G. Egloff, E. F. Nelson and P. Truesdell. *Oil and Gas J.*, 1931, **30** (10), 22.

The gravities of the crude oils from the principal fields, the production level and the date of opening each field are shown in tabular form. Production from Venezuela is chiefly from monoclinal formation, *e.g.*, La Rosa and Lagunillas; whilst that from anticlinal or dome structures is secondary, *e.g.*, Mene Grande, La Concepcion, etc.. Production is from lower Miocene

or Oligocene strata. The analyses of the crude oils, the properties of the distillates and the yield of products obtained from crudes, as reported by the Bureau of Mines, are quoted in the case of oils from the Mene Grande, La Rosa and El Mene fields. The situation, extent and estimated production of these and other fields are given. The chief producing companies are mentioned, and the total production by fields up to the end of 1928, also the annual production of the whole country from 1917/1930 tabulated. Transportation methods and difficulties are referred to, and information given regarding the capacity and situation of the principal refineries. Two experimental cracking tests, using the flashed residue method, have been carried out on fuel oil from La Rosa crude having an A.P.I. grav. of 15·7°, and a visc. Furol at 122° F. of 233 secs. The reaction chamber pressures were 150 lb. and 350 lb. respectively, and the transfer temperatures 925° F. and 915° F. The yields obtained were fairly similar—viz., approx. 27% by vol. of gasoline, 71% of flashed residue and 200 cu. ft. of gas per brl. of charge. The percentages of gas and loss were 1% and 2·6%, the higher loss being experienced at the higher operating pressure. The cracked gasolines possessed the following properties: A.P.I. grav. 57·9°, I.B.P. 92° F., 20% distilling over at 191/184° F., E.P. 397° F., octane number 76/78. The properties of the flashed residues are also stated. The results of commercial cracking operations carried out on La Rosa, La Concepcion and on a mixture of Mene Grande and La Rosa fuel oils are presented in the form of a table. When making gasolines of E.P. 374° F. the yields obtained were 23·22%, 32·04% and 20·9% respectively, whilst for gasolines of E.P. 400° F. the corresponding figures were 25·7%, 35·07%, 22·90%. The operating conditions were: transfer temperatures 896/921° F., pressures 149/163 lb./sq. in. R. A. E.

1057. Refining Crude Oils and the Like. S. T. Henderson (for I. C. I., Ltd.). E.P. 350,494, 12.6.31. Appl., 12.3.30.

The unrefined oil is first treated with a reducing agent, and then given the usual sulphuric acid treatment. It is claimed that better yields and less refining losses are obtained by the use of the preliminary reduction, which may be performed by various agents—*e.g.*, zinc dust or scrap iron with dilute acid. H. C. R.

1058. Treating Petroleum Emulsions. J. Hyman, A. F. Schlandt and Pure Oil Co. U.S.P. 1,811,177, 23.6.31. Appl., 26.3.29.

Petroleum emulsions are broken by introducing a treating agent consisting of an oxidised hydrocarbon motor fuel. The agent may take the form of a gum obtained through the oxidation of hydrocarbon motor fuels produced by vapour-phase cracking and containing a large percentage of unsaturated compounds. W. D. S.

1059. Treatment of Crude Petroleum to Prevent Corrosion. C. D. Gifford, Assr. to Cannon-Prutzman Treating Processes, Ltd. U.S.P. 1,812,629, 30.6.31. Appl., 28.5.28.

This patent describes a continuous method of rendering crude petroleum containing inorganic salts non-corrosive to metals. Steam is injected into a flow stream of the petroleum so that it is heated to a temperature of fluidity and a quantity of water amounting to not more than 6 lb. per brl. of petroleum is added simultaneously. The salts dissolve and the heated petroleum and water are passed through a layer of finely divided diatomaceous earth. The efflux from the layer is separated into an oil and a brine portion by gravity difference. W. S. E. C.

1060. Demulsifying Petroleum. C. P. Dennhardt. U.S.P. 1,814,298, 14·7·31. Appl., 6·9·27.

Apparatus is described consisting of complementary propellers through which petroleum emulsions are pumped. The squeezing action thereby introduced serves to break the emulsion, and the separation is completed in a settling tank. Means are provided for introducing chemicals to further the demulsification if required. H. C. R.

See also Abstracts, Nos. 1069–1100.

Gas.

1061. Factors which Enter into the Design of a Modern Absorber. R. A. Boyd. *Oil and Gas J.*, 1931, **30** (8), 106.

Absorbers are the main controlling factor in the efficiency of a gasoline plant, and the outstanding principles of their design are listed. The minimum diameter of the absorber may be determined on the basis of the velocity of the gas rising through the free space between the plates. This must be kept below that at which serious entrainment occurs. The formula

$$v = \frac{K}{\sqrt{S}}$$

is evolved, where v is the gas velocity, K a constant (calculated to be 0·67), and S the density of the gas under operating conditions. A range of 1 to 2 ft. per sec. is generally considered good practice for gasoline absorbers. The theory is advanced that as an absorber is not efficient as a fractionator, equilibrium conditions are not established during the passage of the oil downwards, so that excess propane, etc., leaves with the enriched oil. Tests have shown rapid absorption of all fractions in top and bottom trays, with light fractions remaining nearly constant in the intermediate trays and only heavier fractions showing gradual absorption throughout. Butane extraction is perhaps the most important function of an absorber, as lighter fractions are not wanted, and if the oil rate is so chosen as to absorb all butane it will be ample to recover the heavier fractions. Tests indicate that with an oil temperature of 80° F. all pentanes and heavier oils can be absorbed if lean oil of 20/27 mm. vapour pressure is used. With a normal type of absorber the maximum recovery of *n*-butane to be expected is 80/85%, no matter how high the oil/gas ratio. Maximum absorber efficiencies are shown when 40/80% of *n*-butane is recovered. Higher *n*-butane recoveries have been obtained, and indicate that the enriched oil leaving the absorber can be supersaturated with *n*-butane as well as with iso-butane and propane. R. A. E.

1062. Recovery of Lighter Hydrocarbons. A. Clarke and H. Butler, Assrs. to United Hydro Carbons Co. U.S.P. 1,813,024, 7.7.31. Appl., 7.8.28.

Hydrocarbons are absorbed from natural gas by an absorption medium, which is subsequently subjected to dry and then to steam distillation. Compression and condensation then follows, and the uncondensed gas is recirculated into the first still, after passing through a stabiliser, the construction of which is indicated. H. C. R.

See also Abstract No. 1132.

Motor Fuels.

1063. Car Manufacturers and Motor Fuels. G. G. Oberfell, *Oil & Gas J.*, 1931, **30** (7), 16.

At the annual meeting of the S.A.E. Dr. Oberfell discussed the question of butane wastage in relation to vapour lock. Using the data obtained by Dr. Bridgeman (Abstract 861) it is shown that an improvement in fuel system design would allow the use of a much larger percentage of butane in the fuel, and that this in turn would give improved starting, accelerating and anti-knock properties. It is estimated that vapour recovery systems installed in refineries could produce sufficient butane to permit the inclusion of 9% in the finished gasoline, and that the natural gasoline which is blended would provide another 3%, making a total of 12% of butane available. However, in 1930 it is estimated that only about 6% was actually used, in order to insure freedom from vapour lock in the average car. In the best type of car tested by Dr. Bridgeman 12% of butane could be used when atmospheric temperatures are not higher than 95° F. On this basis it is estimated that about 4 million gal. of butane per day is wasted at refineries, and that this could be eliminated by an improvement in fuel system design to equal that of the best car tested. In these calculations absence of propane is assumed, and elimination of this product is generally practised at refineries for economic reasons. R. A. E.

1064. Simplify Gasoline Treating Problems. W. T. Ziegenhain, *Oil & Gas J.*, 1931, **30** (8), 29.

The new type of soluble calcium hypochlorite known as H.T.H. (see Abstract 859) is being used in two natural gasoline plants in Seminole, Okla. The gasolines treated do not require a preliminary caustic wash, as they contain no appreciable quantities of hydrogen sulphide, and the process is therefore quite simple. H.T.H. solution is drawn from an open supply tank, contacted in an orifice mixer with the gasoline and the mixture passed to a separating tank. The solution settling out at the bottom is continuously returned to the supply tank and the necessary make-up of H.T.H. added about every hour. The return line discharges below the surface of the solution in the supply tank, and there is sufficient turbulence to dissolve the H.T.H. powder added and to effect uniformity in the strength of the solution. The treated gasoline is tested hourly for control purposes. The relative merits of vertical and horizontal settling tanks are discussed. Both plants treat 8/10 thousand gal. of gasoline per day, and the consumption of H.T.H. is about 1/3 of that of bleaching powder which was formerly employed. There has also been a reduction in corrosion of plant, and in the time devoted to shutting down the plant for cleaning out the orifice mixer, etc. Comparative analyses of H.T.H. and bleaching powder are given. H.T.H. shows far less deterioration on storage; at a temperature of 72° F. only about 2% of the available chlorine is lost in six months. R. A. E.

1065. Question of Fuel Quality. (The Technique of Aero-Engine Fuel Production, with special reference to Ethyl.) F. R. Banks, *Aircraft Eng.*, 1931, **3**, 168–170.

The design of modern aero-engines imposes a very heavy duty on the fuel, the high compression ratios and supercharging both tending to cause detonation with its subsequent evils, so that the fuel is the chief limiting factor in the improvement of engine performance. The author indicates the advantages of fuels made by the addition of Ethyl fluid to straight run spirits

over the usual aviation spirits high in aromatics (*e.g.*, freezing-point is much lower and there is a saving of 0·4 lb. per gal. of fuel carried). The effect of Ethyl fluid (consisting chiefly of tetraethyl lead and ethylene dibromide) varies on fuels of different types. The elimination of detonation reduces the piston temperatures, etc., thus reducing oil consumption, while a good anti-knock fuel permits a higher air/fuel ratio as well as an increase in compression ratio, so that the fuel consumption can also be reduced.

In the United States, the S.30 engine is used to take knock measurements, using a bouncing pin. The conditions of test are described. Knock-ratings are usually expressed on the Octane-Heptane scale—*i.e.*, the percentage of iso-octane (2, 2, 4 trimethyl pentane) in heptane required to match the sample under test. Owing to the high cost of the pure hydrocarbons, two sub-standards are generally used, made by the addition of tetraethyl lead to standard Mid-Continent and Californian spirits respectively.

Tetraethyl lead is the most effective anti-knock substance known, 3 c.c. in a Mid-Continent spirit being equivalent to 40% benzol in the same fuel.

The author considers that modern aero-engines demand a fuel of knock value not less than 80 Octane 20 Heptane. A fuel containing tetraethyl lead has the important advantage that it is equally satisfactory under the high temperature conditions of air-cooled engines and also for liquid-cooled engines.

The paper concludes with a consideration of the effect of ethyl on valves made from various steels. Suitably cooled (*e.g.*, with sodium) cobalt-chrome valves have given good results. Good valve steels are "K.E. 965" and "Era," while nickel chromium steels are very satisfactory. The composition of suitable aluminium bronze valve seat inserts is given and a few comments made on the design, etc., of valve gear.

The author advocates the adoption of a standard fuel for aircraft purposes, and then engine design should be such as to take the maximum advantage of this fuel. C. C.

1066. Motor Fuels. H. Wade, from Standard Oil Co. E.P. 350,438, 5.6.31. Appl., 5.3.30.

Cracked (vapour phase) motor fuel distillates normally tend to deteriorate and develop gum on storage. In order to prevent this an addition is made of less than 0·05% of a substituted amino-phenol (*e.g.*, methylaminophenol or benzyl-amino-phenol). W. H. T.

1067. Fuel for Internal Combustion Engines. L. Mellersh-Jackson, from H/H. Oil Co. A.S. E.P. 350,440, 8.6.31. Appl., 7.3.30.

Aliphatic hydrocarbons for use in internal combustion engines are improved by the addition of aromatic hydrocarbons which have been treated with an equal quantity of nitric acid (below 20° C.), separated from the acid, treated with an equal quantity of sulphuric acid (below 20° C.), followed by another treatment with half the quantity of 70° C. The sulphuric acid is subsequently removed and the product thoroughly washed. W. H. T.

1068. Producing Gasoline from Petroleum Oil. M. L. Chappell, Assr. to Standard Oil Co. U.S.P. 1,811,257, 23,6,31. Appl., 22.1.27.

The vapours from the crude oil are subjected to reflux separation in order to obtain an overhead gasoline stock (F.B.P. below 437° F.). The reflux condensate of higher b. pt. oils is redistilled to give a material of b. pt. between 284° and 437° F. The remaining reflux residue is treated with metallic halide under suitable conditions of temperature and pressure, in order to decompose the oil and form a gasoline stock of b. pt. below 284° F. The three gasoline stocks are finally blended. W. H. T.

See also Abstracts, Nos. 1126, 1127.

Lubricants and Wax.

1069. Distilling Topped Crudes for Lubs. M. B. Miller. *Oil & Gas J.* 1931, **30** (8), 22.

The advantages to be obtained by preparing heavy lubricating stocks by vacuum distillation of topped crude rather than obtaining them from the residual product of distillation are: (1) lower acid usage and treating loss involved in producing oils of equivalent final colour; (2) higher flash-points obtained for oils of similar viscosity; (3) flatter viscosity curve exhibited; (4) elimination of special selection of crudes to be processed. The process is limited in its application to mixed or asphaltic base oils. In treating the overhead stocks it is not necessary to refine the oil to the required colour standard before dewaxing, as in the case of residual stocks. As the cost of centrifuging is less than the cost of refining, and since the losses incurred during the two processes are generally similar, it is often more economical to dewax the overhead stocks before refining.

The vacuum process has been improved to such an extent that the heaviest waxy fractions can be vaporised without sufficient cracking to cause destruction of the amorphous nature of the precipitated wax, which would create difficulty in centrifuging during the dewaxing process. The usual type of vacuum still, in which the oil is passed once through the heating coil and thence to a fractionating column is believed to be adequate for the purpose, provided: (1) the column has sufficient trays of adequate area; (2) the temperature and volume of superheated steam fed at the base of the column is sufficient to compensate for temperature drop in the column. It is advantageous to equip the tower with a side stripping section to assist in bringing the stock to specification and in eliminating crystalline waxes.

An experimental unit is described in which the distillation is carried out in two stages. The topped crude passes to a primary heater, operated at a lower temperature than for single flash distillation, and thence to a fractionating column. The unvaporised oil from the column is reheated to a higher temperature in a second heater, passed to the top of a stripping column into which superheated steam is introduced, and thence to the fractionating column to assist in vaporising the feed. The advantages of this system are: (1) Permits production of heavier fractions by virtue of better stripping; (2) allows better fractionation of vapours; (3) by using a lower temperature in the primary still there is less danger of formation of crystalline waxes in the lighter distillates. The production of a heavier distillate containing heavy amorphous waxes enables greater addition of the lighter distillates to the product to be dewaxed and so further increases the yield. From this unit production has been obtained of distillate oils which were as low in viscosity as 70 secs. at 210° F., but which have been successfully dewaxed and subsequently fractionated into neutral oils and bright stocks. Although no commercial plant has been so operated it would thus appear quite possible to produce an entire range of lubricants by vacuum distillation, centrifugal dewaxing of the whole lubricating stock and subsequent fractionation into grades to meet market requirements. This would be more attractive commercially than dewaxing only the overhead bright stock, as it would eliminate costly low temperature wax pressing of the lighter distillates. R. A. E.

1070. Properties and Refining of Paraffin Wax. M. Bestushew. *Erdöl und Teer*, 1931, **7**, 255–257, 270–274, 301–306.

Many views have been advanced to account for the difference between amorphous wax separated from distillates by centrifuging and crystalline wax. The author favours the view of Sachanen, Wassiliew and Sherdewa,

who suggest two series of high-melting compounds are present—paraffin and ceresine. The latter series has (for equal melting-points) higher boiling-points, molecular weights and tendency to decompose on heating.

Micro-photographs are given showing the difference in structure between ceresine, mixtures of paraffin and ceresine, and paraffin, the latter having a distinct "banded" structure. The presence of ceresine makes filtration far more difficult on account of the smaller crystals then formed in the wax. Very rapid cooling will tend to give paraffin a ceresine-like structure. Good rectification is necessary in the distillation of paraffin, otherwise ceresine is liable to come over in the light machine-oil fraction, necessitating a second or even third distillation. The effect of cracking-distillation is to promote the decomposition of ceresine; with good rectification it is seldom necessary. The filtration process was investigated in detail in a specially jacketed and cooled filter-press. The success of the process depends on the temperature, the pressure and the time of pressing; the temperature being the most critical of these factors. The size, etc., of the plates also affects the composition of the press-cake. Viscosity data on various oils are also given from 50° C. to —15° C. The following recommendations are made for good filtering: (1) There should be no ceresine in the paraffin distillate; (2) The viscosity should be as low as possible and not over 2°E at 50° C.; (3) wide fractions containing light oil should be used; (4) the filtration should be carried out at as high a temperature as possible, which is best determined experimentally for the distillate to be treated; (5) filter-plate spacing greater than 20 mm. is not advisable; (6) pressures up to 50 atms. may be used, and, in effect, increase the capacity of the press.

The sweating-process is also considered, the effects of temperature regulation being important. A rise of 1° C. per hr. is recommended. The oil content of finished wax determines its stability to light, oil-free waxes being light-stable, those with 0·5% oil slowly turning yellow and those with over 3% discolouring very rapidly. A method is described for estimating the oil content from the penetration as determined with the Richardson penetrometer.

E. B. E.

1071. LUBRICANTS. W. W. Triggs. E.P. 351,041–2, 18.6.31. Appl., 18.3.30.

The viscosity of a mineral oil is raised to any desired degree by adding 6–9% of a metallic salt of soap-forming fatty acid to the oil, heating to 350° F. with stirring and cooling in order to thicken. An oil treated in this manner to give a product ranging through jelly-like to soap-like consistencies may be mixed with a vegetable oil treated in the same way (E.P. 349,684). In this way a solidified lubricant is obtained which will flow when a certain desired temperature is exceeded and which will, at the same time, have the property that it is capable of becoming more fluid and better capable of acting as a lubricating medium when pressure is applied. W. D. S.

1072. REFINING OILS. A. R. Moorman, I. C. Carpenter and Contact Filtration Co. U.S.P. 1,809,862, 16.6.31. Appl., 4.1.29.

Acid treated lubricating oil is mixed with an adsorbent partially spent in decolourising neutralised oil, and the temperature raised to 250° F. This effects neutralisation. The mixture is then cooled to below the temperature of rapid oxidation of the oil and the neutralised oil separated from the spent adsorbent. Decolourisation is effected by treating with fresh adsorbent at 250° F. This partially spent adsorbent is then used for the initial process.

W. D. S.

1073. Refining Oils. W. T. Bryant and W. A. Williams. U.S.P. 1,812,923, 7.7.31. Appl., 30.8.27.

Oil is refined by acid treating, removing the sludge, and heating to above 212° F. and below its flash point. The oil so heated is delivered to a clay treating chamber where it is maintained at an elevated temperature. The clay is added at a temperature such that reaction between the clay and oil is instantaneous. The oil is immediately conducted from the chamber through a continuous filtering means and moisture removed by volatilization of the water in a vacuum. W. D. S.

1074. Manufacture of Lubricating Oils. W. T. Bryant and W. A. Williams. U.S.P. 1,812,924, 7.7.31. Appl., 30.8.27.

The crude oil is distilled with a small quantity of dilute caustic alkali solution until the remaining charge of alkali and oil in the still is only 5%. The lighter fractions are removed from the distillate and the reduced distillate chemically treated with acid of less than 98% conc. The sludge is removed and the oil decolourised with absorbent clay. W. D. S.

1075. Treatment of Petrolatum Stocks. D. L. Souther, W. A. Gruse and Gulf Refining Co. U.S.P. 1,813,327, 7.7.31. Appl., 5.6.28.

Petrolatum stock is separated into resins and asphalts, wax and oil of low wax content and low carbon residue test, by the following process. The stock is dissolved in butyl alcohol at the temperature of solution (180° F.), the solution cooled sufficiently to separate contained resins and asphaltic material (140° F.), but not so low as to remove wax. The separated material is removed and the residual liquid cooled in a plurality of stages, wax being removed at each stage. Acetone is added at the last stage (32° F.). The acetone and butyl alcohol are distilled off and there remains a residual heavy mineral oil of low wax content and low carbon residue test.

W. D. S.

See also Abstracts, Nos. 1129–1131.

Fluxes and Asphalts.

1076. Process for Treating Aqueous Bituminous Dispersions. H. D. Elkington. E.P. 350,040, 11.6.31. Appl., 11.3.30.

A process is described for the preparation of dispersions, especially those of asphalt, tar and paraffin, for use in road construction, for surface dressing of roads and as protective coatings.

The aqueous bituminous dispersions are blended with alkali metallates which react with the atmospheric carbon dioxide to form coagulating agents; these promote the coagulation of the dispersed particles. W. S. E. C.

1077. Bitumen Emulsions. W. A. Walmsley and R. H. Thompson (for Thomas Ness, Ltd.). E.P. 350,744. 18.6.31. Appl., 24.6.30.

The invention relates to the production of aqueous bitumen emulsions, using as emulsifying agents the alkali salts of carbozol, of α- and β-naphthates, of pyrogallic acid, or of resorcinol. The proportion used is from 0·25% to 1·0% of agent to bitumen, and rosin soap or the like may also be added.

H. C. R.

See also Abstract No. 1133.

Special Products.

1078. Methanol Anti-freeze and Methanol Poisoning. W. P. Gant, H. H. Schrenk and R. R. Sayers. *Ind. Eng. Chem.*, 1931, **23**, 551–555.

Although the use of methanol as an anti-freeze for car radiators is not entirely new, it is only recently that its low cost and the ease with which it is obtainable have brought it into general use. It is estimated that during the past season 4,000,000 gals. were consumed for this purpose in the States. Physically and chemically, methanol is quite a satisfactory anti-freeze. There is, however, much conflicting data concerning its poisonous properties, and the present investigation is being carried out with the object of evaluating the dangers encountered in its manufacture and use. The investigation is still incomplete, but so far experiments with animals have shown that although there is a range of concentration of the vapour in air and periods of exposure which cause no apparent harm, there are conditions which do definitely produce methanol poisoning. These conditions are much less severe than could accompany the use of methanol as an anti-freeze. It is suggested that methanol should be coloured, given an unpleasant taste and marketed in special containers marked "poison."

S. F. B.

1079. Flame-Pressure Process for Carbon Black. A. W. Francis. *Ind. Eng. Chem.*, 1931, **23**, 612.

The best grades of carbon black are made by burning hydrocarbon gases with a continuous flame. The yields are seldom over 3% because the upper inflammability limits of the hydrocarbons are so low that an excess of oxygen is always present to consume most of the carbon. Stifling the flames is futile.

Since an increase in pressure raises the upper inflammability limits, much higher yields of carbon black can be obtained by burning hydrocarbon continuously in an atmosphere of compressed air. Yields of high-grade carbon black have been trebled at 100 lb. pressure in small scale equipment, and computations have been made indicating that this can be increased much more. The process also permits utilisation of the heat of combustion of the gas.

W. D. S.

1080. Insecticidal and Fungicidal Compositions. Standard Oil Dev. Co. E.P. 350,897, 15.6.31. Appl., 15.2.30.

An insecticidal, insect-repellent or fungicidal composition is made from petroleum white oil (0·25% to 1%), an extract of plant belonging to the class known as "fish poisons" (derris or cubé extract), water, and an emulsifying agent. The latter may be a soluble alkali sulphonate derived from petroleum oil. Pyrethrum and an anti-oxidation catalyst may be additional ingredients.

W. D. S.

1081. Regeneration of Non-Aqueous Solvents, such as those Employed in the Dry Cleaning Industry. W. Y. Agnew. E.P. 350,904, 10.6.31. Appl., 10.3.30.

A treatment for impure solvents used in dry cleaning processes is outlined. The suspended material is flocculated by the use of concentrated acid and is then removed by sedimentation, centrifuging or filtration through an alkaline bed. The use of less than 0·05% of sulphuric, hydrochloric or phosphoric acids with respect to total solvent is advocated. H. C. R.

1082. Treating Gas Mixtures containing Saturated Hydrocarbons. Soc. d'Etudes Scientifiques et d'Entreprises Ind. (Assignees of Edmond Voituron). E.P. 352,200, 9.7.31. Appl., 29.7.30. Conv. (Germany), 29.7.29.

The invention relates to a process of treating hydrocarbons with steam and oxygen at a temperature of 1300° C. at a space velocity through the reaction chamber of 100,000 vols. per hr. per unit vol. of reaction chamber. It is claimed that considerable amounts of acetylene are formed in addition to carbon monoxide and hydrogen. H. C. R.

See also Abstract No. 1128.

Refining and Refinery Plant.

1083. Explain Various Clay Recovery Methods. F. W. Zublin. *Oil and Gas J.*, 1931, **30** (6), 22.

The recovery of coarse clays, such as those used for vapour phase treatment, is relatively simple, as it can be achieved by burning. The recovery of fine clays, such as those used in contact processes, is relatively difficult, and other methods must be employed. The first operation is to remove excess oil and the second to remove adsorbed matter. Reference is made to a number of patents dealing with the first operation. The forces operating to cause adsorption are (1) mass attraction, or non-polar force, which depends chiefly on the size of the molecules to be adsorbed; (2) electrical, or polar force, which varies according to the chemical composition of the reacting medium. These forces are both inversely proportional to the square of the distance from the mass centre. The action of solvents is therefore limited to the point at which solution pressure and adsorption forces are equal, unless the solvent has considerable surface affinity to the clay, in which case the material originally adsorbed may be gradually displaced. Heats of wetting were considered by Gurwitsch to be a quantitative measurement of adsorption forces, and this has been confirmed by Bosshart and Wilde. The results obtained by Gurwitsch using three types of clay show that unsaturated compounds and compounds with a hydroxyl radicle possess the highest heats of wetting. For hydroxyl compounds chemical similarity and consequent polar attraction is responsible, and the smaller the balance of the molecule the higher the heat of wetting. For non-polar adsorption the larger the molecule the greater the heat of wetting, polymerisation being responsible for the high value of unsaturated compounds. In the removal of adsorbed material, therefore, highly polar compounds of low mol. wt. or non-polar compounds of high mol. wt. are most efficient. In removing colouring matter from oils polar substances of low mol. wt. must be avoided, but hydrocarbons of low mol. wt. are most suitable. Gurwitsch found a mixture of methyl or ethyl alcohol and benzol effective for revivifying clays; by wetting the spent clay with naphtha and alcohol and extracting in a soxhlet extractor with benzene he was able to remove 99·5% of the adsorbed resins. Reference is made to patents in which compounds containing hydroxyl groups are utilised, and, it is thought that triethanolamine should be suitable as a displacing solvent. The reasons why water and caustic soda solution are unsuitable are given. Reference is made to a patent in which the use of a non-polar solvent, such as kerosine, at high temperatures (500° F. or over) is proposed. Clays which have been used for treating acid oils present great difficulty in recovery, but Rosenbaum claims to have overcome this problem by flushing the clay with liquid sulphur dioxide. R. A. E.

1084. ANHYDROUS LIQUID SULPHUR DIOXIDE. L. B. Hitchcock and A. K. Scribner. *Ind. Eng. Chem.*, 1931, **23**, 743–749.

Sulphur dioxide owes its present commercial importance primarily to its physical properties rather than to its better known chemical characteristics. Thus its ability to vaporise at 14° F. with the absorption of 167·1 B.Th.U. per lb. in part explains its utility in refrigerating plants while its high gravity, 1·49, considerably adds to its value in the counter current extraction of sulphur compounds, aromatic hydrocarbons, etc., from petroleum distillates. In both fields it is essential that the liquid used should be substantially anhydrous if corrosion is to be avoided, and a maximum of 25 parts per million of water corresponding with sulphur dioxide 99·9975% pure is demanded. At the Virginia Smelting Company, East Norfolk, Va., a plant has been developed capable of providing liquid sulphur dioxide containing only 15 parts per million. Although the conversion of crude sulphur to sulphur dioxide appears to be a simple process, the plant required is relatively elaborate and involves a large capital outlay. Labour charges are however very low owing to the design and close technical control.

The sulphur is fed into a Glens Fall rotary burner, care being taken to control the air admitted so that only traces of sulphur trioxide or unburnt sulphur are carried to the next stage. Traces of unburnt sulphur however cannot be entirely prevented, and these are oxidised in a secondary combustion chamber to which a further supply of air is admitted. The crude gas is next delivered by a Sturtevant fan to the scrubbers, a series of five packed towers through which sea water flows in counter current flow, the gas leaving the last scrubber consists of nitrogen and 0·02–0·01% sulphur dioxide. The water leaves the first tower saturated with sulphur dioxide and containing ordinarily 1–2%. The absorption water is next stripped in a tower equipped with a barometric leg by means of low pressure steam, water vapour and sulphur dioxide at approximately 95° F. leaving the top, while water at 135° F. and 25·5 inches of vacuum containing only 0·01% sulphur dioxide leaves the bottom. This waste water is extremely corrosive, and as no material has yet been found suitable for a recovery plant, even if the necessary cost warranted it, it is discarded. The warm gas passes by way of a surface condenser to the compression plant, any condensate formed being allowed to flow to waste through a 29-foot head. The gas from the first compressor is discharged to the first watercooler, the condensate flowing through a leg 20 ft. high to the atmosphere. The second stage compressor brings the gas up to approximately atmospheric pressure, the watercooler draining through a simple trap. A two-stage Worthington tandem compressor boosts the gas up successively to 25 and 120 lb./sq. in., a suitably trapped watercooler being placed between these stages; approximately 1% of water remains in the gas when it enters the buffer drum before the drying unit. This unit contains the standard features of a recycling system as found in all processes involving the removal of a product by compression and cooling. In addition it has a fractional condensation unit, the design of which is one of the features of the plant. The gas leaving the drying unit contains only 10 parts per million of water; it is liquefied by passage through a condenser in the usual way and stored in one of the two 96,000 lb. storage tanks provided. The condensate from the drying unit contains between 10 and 20% of water, and is passed to a reclaiming tank at any pressure varying from 26 in. of vacuum to atmospheric. The sulphur dioxide at once flashes off and is recompressed by a two stage Worthington compressor, any liquid being removed between the stages by an interstage cooler. The uncondensed gas passes back to the buffer drum for drying.

S. F. B.

1085. Apparatus for Carrying Out Gas Reactions at High Temperature. I.C.I., Ltd. T. S. Wheeler and W. B. Fletcher. E.P. 349,958, 27.5.31. Appl., 27.1.30.

Endothermic gas reactions are carried out at temperature over 1000° C. and at high space velocities by passing the gas through unpacked reaction chambers which are long, deep and very narrow and are sandwiched between long and deep heating flues. The chambers have a width of the order of 1 in. to 2 in., and are made of refractory material capable of resisting a temperature of at least 1000° C. The desired reactions take place in the chambers, and the products pass through recuperators where their surplus heat is utilised in preheating further quantities of reactants. W. D. S.

1086. Defrosting Heat Exchangers in Apparatus for the Liquefaction of Gaseous Mixtures. L'Air Liquide S.A. E.P. 350,057, 11.6.31. Appl., 14.3.30.

In apparatus for the partial liquefaction or separation by liquefaction of gaseous mixtures, defrosting is constantly necessitated in the heat exchangers. If two exchangers are used in such a manner that one is defrosted while the other is in normal operation, the operation is facilitated. The whole or part of the gas remaining after partial liquefaction which has passed through the exchanger in normal operation, is passed respectively in the same paths through the exchanger to be defrosted. The defrosting is then achieved by heating the gases. W. D. S.

1087. Vaporisation of Heavy Hydrocarbons. A. Joseph, Assr. to Cie Int. Fabrication des Essences et Petroles. U.S.P. 1,806,997, 26.5.31. Appl., 14.7.29.

The invention relates to an apparatus for the vaporisation of liquid or pasty heavy hydrocarbons with a view to their subsequent conversion into light hydrocarbons. The plant consists essentially of a vertical cylindrical chamber surrounded by a heating jacket, the heating gas entering at the bottom of the jacket and leaving at the top. The top portion of the inner chamber contains a series of baffle plates while the lower two-thirds is packed with porous material. Oil (and water or steam) are admitted at the top of the chamber, and the former is partially vaporised on coming into contact with the hot baffle plates. The vaporisation is continued and completed on the bed of porous material, where deposition of coke, etc., occurs in a very sub-divided form, which permits of its subsequent perfect elimination by combustion by means of a current of air or oxygen. W. H. T.

1088. Condensing Gasoline Vapours. F. W. Isles, Assr. to Standard Oil Dev. Co. U.S.P. 1,809,167, 9.6.31. Appl., 25.1.27.

The patent describes a method for condensing light petroleum vapours mixed with incondensable gas from absorption stock obtained from a refinery gasoline recovery plant. The vapours are cooled to a condensing temperature in the presence of benzol when a benzol-petroleum blend is formed and the vapours are substantially removed from the incondensable gas. The blend is collected and the gas withdrawn. The benzol blends produced are suitable for mixing with heavy naphtha to give very good motor gasoline. The yield of gasoline constituents obtained by this method is usually increased by 30% or more as compared with that obtained by simple condensation.

The process can also be applied in charcoal absorption plants for the recovery of gasoline from natural gas; in this case the benzol is brought into contact with the vapours expelled from the saturated charcoal.

W. S. E. C.

1089. Refining Hydrocarbon Oils. A. Lachman. U.S.P. 1,809,170, 9.6.31. Appl., 8.5.29.

An apparatus is described for the contacting of vaporised mineral oils with a water solution of a mineral salt—*e.g.*, zinc chloride maintained in solution by the addition of the requisite amount of water or steam. By using concentrations of the metallic salt as high as 50–85%, the condensed vapours yield a refined oil. H. C. R.

1090. Treating Hydrocarbon Oils. J. C. Black and M. L. Chappell. U.S.P. 1,810,068, 16.6.31. Appl., 31.1.28.

A process of treating petroleum distillates containing cracked gasoline with sulphuric acid at low temperatures is claimed. The charging stock is first cooled by the outgoing treated gasoline, and is then blended with approximately 10% by volume of liquefied hydrocarbon gas, such as the liquefiable portion of the gases from a cracking process. The blend is treated with acid of not less than 76% strength in two stages, at about 17°F., the acid tar separated and the dissolved gases removed by suitable heating. By this means it is claimed that while objectionable sulphur compounds and unstable hydrocarbons are attacked, the hydrocarbons having valuable anti-knock properties are unaffected and the treating loss is much reduced. A. J. G.

1091. Apparatus for Catalytically Treating Hydrocarbon Oil. Gustav Egloff to Universal Oil Products Co. U.S.P. 1,810,648, 16.6.31. Appl., 16.8.22. Renewed 1.9.28.

An apparatus is described for the catalytic decomposition of hydrocarbon oil under suitable conditions of pressure and temperature with a substance such as aluminium chloride. The apparatus consists simply of a still, provided with the necessary inlets and outlets for oil and catalyst, connected to suitable condensing equipment. An essential feature is the provision in the vapour space of the still of a water-cooled revolving drum which, it is claimed, causes condensation of the catalyst and effects partial dephlegmation of the vapours. A. J. G.

1092. Decolorising Oils. R. C. Osterstrom and the Pure Oil Co. U.S.P. 1,811,185, 23.6.31. Appl., 18.6.28.

A continuous method of purifying mineral oils with adsorbent clays is described. The oil and clay are mixed and heated to a temperature above that at which the oil under treatment vaporises, but below the cracking temperature. The evolution of oil vapours is prevented by the application of pressure, the latter being sufficient to keep the oil substantially in the liquid phase. The pressure on the flowing stream of oil and clay is then reduced, when the oil vaporises. The vapours are brought into intimate contact with absorbent clay for the desired period; by further reduction of the pressure the desired lighter and purified fractions may be separated from the clay. W. D. S.

1093. Oil Fractionating Apparatus. C. L. Smith and C. B. Watson. U.S.P. 1,811,189, 23.6.31. Appl., 28.2.28.

A fractionating apparatus capable of giving very effective heat interchange between the arising hot oil vapours and the descending liquid condensate is described. It consists of a shell with vapour inlet at the bottom and outlet at the top between which inlet and outlet is a fractionating section. The latter comprises a plurality of superposed groups of nested trays arranged so as to produce tortuous vapour passages between the trays. The trays include a trough for the reception of liquid condensate, and above the trays are apertured distributing pans. W. D. S.

1094. Refining Oils with Metallic Halide. J. H. Osmer and Standard Oil Co. U.S.P. 1,811,243, 23.6.31. Appl., 25.2.27.

A heavy hydrocarbon oil which it is desired to refine is mixed with a lighter oil which has previously been purified by treatment with a metallic halide. This mixture is contacted with a metallic halide at a temperature insufficient to cause any substantial conversion to lower boiling oils—*e.g.*, about 200–250° F. The metallic halide, purified heavy oil and light low boiling oil are separated. The heavy oil may require further purification by means of acid or clay. W. D. S.

1095. Emergency Relief with Remote Control. A. T. Dudley. U.S.P. 1,811,351, 23.6.31. Appl., 21.6.28.

In a pipe still with tubes fitted in series and so arranged that the top pipes where the oil enters are heated by convection, while the lower pipes before the oils egress are heated by direct radiation, a U bend connecting the last pipes of the convection section is brought outside the brickwork. A side tube is fitted to the U tube with two valves, one in the side tube while the other is in the lower limb of the U, between the side tube and radiant section. In the event of trouble in the radiant section, the oil is diverted down the side tube by remote control of the valves. A. R. S.

1096. Oil Refining System. D. Pyzel. U.S.P. 1,811,645, 23.6.31. Appl., 27.12.20.

Vapours from a still are passed into a bubble tower where the heavier hydrocarbons likely to emulsify with water are condensed. The lighter hydrocarbons then pass into direct open contact with water, which may be impure or salt, for condensation. Thus condensation of all products is completed without formation of fixed emulsions. A. R. S.

1097. Dephlegmating Apparatus. G. Egloff and H. P. Benner. U.S.P. 1,812,261, 30.6.31. Appl., 28.6.20.

In an apparatus for cracking hydrocarbons, a vertically elongated dephlegmator has a series of baffles set therein vertically disposed with respect to each other. The dephlegmator has a vapour inlet at the bottom and also a liquid outlet adjacent to this. Intermediate with the baffles are a set of outlet pipes, each with an independently operated valve, so that the vapours can be drawn off through any desired conduit. The apparatus works at a pressure greater than atmospheric. A. R. S.

1098. Refining Hydrocarbons. E. W. Isom, Assr. to Sinclair Refining Co. U.S.P. 1,812,439, 30.6.31. Appl., 29.2.28.

This invention relates to improvements in the refining of cracked (vapour phase) gasoline, in which a vapour mixture including the gasoline product (50–75%) is passed through an adsorptive catalyst. The gasoline is fractionated from the mixture and the residue returned to the cracking operation. W. H. T.

1099. Refining Hydrocarbons. W. Mendius, Assr. to Sinclair Refining Co. U.S.P. 1,812,446, 30.6.31. Appl. 10.2.28.

An apparatus is described for contacting hydrocarbon vapours with an adsorptive catalyst. It comprises a receptacle and means for supporting a charge of catalyst therein so that the latter will extend above the auxiliary connections.

Hydrocarbon vapours are supplied to one end of the receptacle and removed from the other, a series of auxiliary vapour connections being provided having valves arranged along the normal path of vapour travel through the adsorptive catalyst. W. H. T.

See also Abstract No. 1064.

Cracking.

1100. Crack Crude Direct with Dubbs Unit. E. F. Nelson and G. Egloff. *Oil and Gas J.*, 1931, **30** (9), 20.

The latest development in cracking is to take crude oils of high gasoline content and crack them directly, thus obtaining high yields of anti-knock gasoline and eliminating entirely skimming operations and the production of straight-run gasoline. This system gives a larger yield of anti-knock gasoline than is obtainable by methods of partial cracking. When operating the plant in this way, crude oil and condensate drawn continuously from the bottom of the fractionating column are pumped to the heating coil. The heated oil passes to a reaction chamber maintained under pressure, and from thence to a flash chamber, in which a lower pressure is maintained. The residue is continuously drawn off from the bottom and is cooled and discharged to storage. The vapours pass to a fractionating column where separation into gas and reflux condensate is obtained. The vapours from the column pass through a condenser coil and thence to a distillate receiver, from the bottom of which the required gasoline is discharged to storage. The incondensable gas from the distillate receiver is scrubbed to remove gasoline fractions. The reflux condensate from the fractionating column is returned to the heating coil mixed with incoming crude.

The following data is presented on Joiner crude: (1) Direct cracking yields 70·9% of gasoline having an octane number of 70, and 7·1% of gas and loss; (2) cracking the topped crude and blending the gasoline with the straight-run product yields 70·9% of gasoline having an octane number of 66, and 5·6% of gas and loss; (3) cracking both the topped residue and the straight-run gasoline yields 66·4% of gasoline having an octane number of 72, and 9·1% of gas and loss. It is estimated that a plant having a capacity of 1000 barrels per day for cracking topped crude could treat 1300 barrels a day of crude containing 35/40% of straight-run gasoline. A chart is presented to show the average T.E.L. requirements of gasolines of varying octane numbers in order to equal ethyl standard. This relationship may vary somewhat for any particular gasoline on account of variation in lead susceptibility.

Complete information is given of the experiments carried out in the treatment of Joiner crude by the three different methods mentioned, and the properties of the gasolines, topped residue and flashed residues obtained as intermediate or final products are detailed. Gasolines from Joiner crude are easily refined by acid, soda and water or by treatment in the vapour phase.

R. A. E.

1101. Cracking to Improve Pennsylvania Gasoline. F. R. Staley. *Petr. Eng.*, 1931, **2** (1), 29.

The low octane number of the gasoline from Pennsylvania crude has led to the installation of cracking units for the production of high-grade gasoline from the kerosine and gas oil fractions.

The Emlenton Refining Co. has recently installed a Dubbs cracking unit with a capacity of 750 brls./day, producing gasoline of octane number over 70. The cracked gasoline is refined in the vapour phase by two Gray towers, using 30–60 mesh Georgia clay, the resulting product being sweet, non-corrosive and stable to sunlight, without further treatment. A feature of the cracking plant is the Alcorn furnace, the efficiency of which is very high,

as very little excess air is required and heat distribution is very uniform owing to the arrangement of the tubes and method of firing and general furnace design.

Details of the general refinery equipment and methods of production and refining are given. C. L. G.

1102. Process for Cracking Oils, Tars and the Like. S. Seelig. E.P. 348,242, 14.5.31. Appl., 20.2.30.

The invention relates to special forms of vapour chambers wherein the cracked product from a cracking coil is stripped of its light products. The method adopted is to pass the hot products from the cracking coil through tubes situated at the bottom of the vapour chamber, thence to perforated tubes in the upper part of the chamber so that the oil is sprayed on to the lower tubes. It is claimed that it is possible to make use of the temperature difference between released and unreleased oil, as a considerable temperature reduction takes place when vaporisation occurs.

A second form of the apparatus is shown having a vaporiser with separate heating. R. P.

1103. Cracking Hydrocarbons. Sinclair Refining Co. E.P. 348,764, 21.5.31. Appl., 11.3.30. Conv. (U.S.A.), 15.5.31.

The process consists of a combined mixed-phase, high-pressure cracking operation, a vapour-phase, low-pressure cracking operation and a direct coking operation. By carrying out the combined operation to produce coke instead of tar, higher yields of gasoline are obtained and the residue is reduced to a more merchantable form. The process is carried out by passing the vapours from the coking still beneath the surface of a liquid body of oil in a scrubbing vessel, and the oil from the vessel is passed back to the coking still. The vapours are subjected to fractionation in two towers. The reflux oil from the first column is heated in a heating coil under high pressure, whilst the reflux from the second column is subjected to a heat treatment at low pressure. The products from both heating coils are allowed to expand into the coking chamber. Gasoline is condensed from the vapours leaving the second fractionating tower. R. P.

1104. Cracking Hydrocarbon Oils. E. W. Isom. U.S.P. 1,807,241/2, 26.5.31. Appl., 27.7.27, 27.12.29.

The charge of oil is circulated from a supply drum, through heating coils and back to the supply drum. A bed of absorbent material is placed in the drum and extends entirely across the path of the circulating oil. The maintenance of a pressure drop through the filtering bed is an important part of the invention, and it may be obtained by taking vapours from above the bed under pressure and vapours from beneath the bed at a lower pressure. The vapours may be separately subjected to refluxing operation and the reflux condensate returned to the circulating oil. Oil may be returned above or below the filtering bed and pitch-laden residue may be withdrawn above or beneath the filtering material. R. P.

1105. Cracking Hydrocarbons. E. C. Herthel. U.S.P. 1,809,297, 9.6.31. Appl., 8.2.30.

A cracking plant is described consisting of two series-connected batteries of vertical heating tubes in a vertical heating flue, the heating gases entering and traversing the flue transversely to the direction of travel of the hydrocarbon oils. A reflux tower and a vapour separator and the usual condensing systems complete the plant. H. C. R.

1106. Cracking Hydrocarbons. E. C. Herthel, Assr. to Sinclair Refining Co. U.S.P. 1,810,048, 16.6.31. Appl., 7.3.29.

An improved form of plant for carrying out a combined vapour-phase cracking and coking operation is described, in which the oil to be coked is heated by direct contact with the vapours from the vapour-phase cracker. The combined vapours are dephlegmated in a baffled tower and then fractionated in a suitable column. The reflux condensate from the fractionating column is used in the baffled tower as a refluxing medium and is then fed to the vapour-phase cracker. A. J. G.

1107. Hydrocarbon Oil Conversion. J. C. Morrell and W. F. Farragher, Assrs. to Universal Oil Products Co. U.S.P. 1,810,673, 16.6.31. Appl., 1.2.29.

A process is described in which suitable charging stock is heated to a cracking temperature under super-atmospheric pressure and is discharged into a large drum at a reduced pressure, thereby effecting flash evaporation. The evolved vapours are dephlegmated in suitable equipment; part of the reflux condensate from the dephlegmator is combined with the charging stock and part is subjected to cracking conditions of pressure and temperature in a separate heating zone and is then again discharged into the flash evaporator.

It is claimed that by this separate cracking of the reflux condensate the products are improved and the quantity of the coke formed is decreased. A. J. G.

1108. Method of Converting Oils in Vapour Phase. C. R. Wagner. U.S.P. 1,811,194, 23.6.31. Appl., 21.8.29.

Hydrocarbon oil is heated to vaporisation and the vapours separated and passed as vapour into the tubes where it is cracked at 1000–1100° F. The vapours then pass through a section where substantially only endothermic cooling occurs to not less than 1000° F. From this section the vapours are again heated to cracking temperature without interruption of endothermic reactions. From here the vapours are condensed. A. R. S.

1109. Method of Treating Petroleum Oil. C. B. Watson. U.S.P. 1,811,195, 23.6.31. Appl., 9.2.27.

A process is claimed whereby oil is cracked in the vapour phase to produce anti-detonating fuels, and consists in vaporising a stream of high boiling-point oil, heating the vapours to a temperature of between 750 and 900° F., then injecting the vapours at a high velocity into reaction zone against a stream of superheated steam having a temperature of 1000 to 1500° F.

The vapours are withdrawn from the reaction zone and suddenly cooled. The light vapours are then fractionated to obtain the desired motor spirit. R. P.

1110. Cracking of Hydrocarbons. E. W. Isom. U.S.P. 1,811,272, 23.6.31. Appl., 30.6.24.

The invention claims improvements in the method of pressure distillation, including a cyclic process in which tar is drawn off from the still and treated to remove tarry constituents. The tar is treated for the separation of carbonaceous matter by admixture with fine Fuller's earth or similar material. After settling, the oil can be returned to the process.

Apparently the Fuller's earth particles act as nuclei about which tarry or carbonaceous particles collect, leaving the oil relatively free from these constituents. R. P.

1111. Process for Producing Low Boiling-Point Oils. C. P. Dubbs. U.S.P. 1,811,308, 23.6.31. Appl., 17.5.20. Renewed, 24.4.28. Divided and this appl. filed, 5.5.28.

The salient objects in the invention are to provide a process in which the temperature of the dephlegmating agent is controlled so that either a large or a small proportion of the vapours are condensed; and in which the temperature of the dephlegmating stages may be varied to produce the greatest possible yield and more uniform product; and that any particular fraction may be extracted from the oil without redistillation. R. P.

1112. Process for Treating Petroleum Oil. C. P. Dubbs. U.S.P. 1,811,617, 23.6.31. Appl., 31.7.22.

Oil is cracked by passing through a heating zone, whence it flows to a series of reacting vessels, where vaporous products are removed for condensation and collection.

The heaviest unvaporised oil is removed from the system, but the lighter unvaporised oils from subsequent chambers are returned for a further treatment in the heating zone, the heavier unvaporised oil from the reheating being removed from the system as before in a subsequent chamber.

A. R. S.

1113. Apparatus for Hydrocarbon Oil Conversion. O. H. Fairchild. U.S.P. 1,811,621, 23.6.31. Appl., 10.10.27.

The cracking apparatus consists of carborundum piping superimposed on a furnace, the joints in the piping being protected against leaks by the introduction of grooved baffles. A. R. S.

1114. Hydrocarbon Oil Treatment. D. Pyzel. U.S.P. 1,811,646, 23.6.31. Appl., 29.6.27.

In a cracking process, the oil is heated to cracking temperature in a coil, from which it is delivered into a reaction zone where substantial vaporisation occurs. The oil is delivered into this zone under the surface of an oil layer in which the unvaporised oil remains. The oil in this layer is kept in slow movement and a draw-off is provided to keep the bulk of oil relatively constant.

A. R. S.

1115. Apparatus for the Conversion and Transformation of Hydrocarbons, Liquids and Fluids. J. H. Adams. U.S.P. 1,811,834, 30.6.31. Appl., 12.1.29.

High-boiling hydrocarbon oil is cracked by passing through an elongated, tubular coil consisting of a number of pipes connected in series, the oil being forced through the coil under pressure, into a large unheated collecting chamber, which causes settlement of carbon and heavy oil.

The vapours pass upwards through a tray where they are cooled by heat exchange with accumulated oil before passing out of the chamber, which is also operated under pressure. A. R. S.

1116. Apparatus for Heating Petroleum. A. Oberle. U.S.P. 1,812,217, 30.6.31. Appl., 19.4.23.

The invention relates to improvements whereby oil is preheated in a rectifier and thereafter subjected to cracking. The vapours are dephlegmated and passed through the incoming raw stock, in order to improve the quality of the resulting distillate. A process is claimed, consisting of introducing the oil into a pool, positioned in a heating means and maintained at a controlled level, passing the oil to a cracking zone and raising the oil to a cracking temperature, passing the vapours to a dephlegmator and returning the reflux oil to the oil pool and condensing the vapours. R. P.

P1117. Process of Treating Hydrocarbon Oil. J. D. Seguy. U.S.P. 1,812,308, 30.6.31. Appl., 19.12.28.

The object of the invention is to produce a desired yield of gasoline while at the same time limiting or preventing the formation of carbon when handling heavy oils.

A process is claimed whereby heavy oil is subjected to a cracking temperature under pressure. Thereafter the vapours are separated from the heavy residue, which is removed from the cracking zone before it contains objectionable amounts of sludge and coke. The residue is mixed with distillate bottoms to cool it, and thereafter the pressure is reduced to cause partial vaporisation. R. P.

1118. Conversion of Hydrocarbons. E. Gallo and H. Zorn, Assrs. to I. G. Farbenind A.-G. U.S.P. 1,812,398, 30.6.31. Appl., 19.12.28.

The conversion of hydrocarbons of high molecular weight into those of low molecular weight is effected by passing the vapours of the raw material at a sufficiently high temperature in association with inert gases over a catalyst comprising a salt of metaphosphoric acid. W. H. T.

Hydrogenation.

1119. Producing Low Boiling Oils from Higher Boiling Oils. Standard Oil Dev. E.P. 348,889, 21.5.31. Appl., 20.5.30. Conv. (U.S.A.), 4.6.29.

The process comprises destructively hydrogenating heavy oil in the liquid phase at a relatively low temperature (750–925° F.), whereby low boiling and intermediate products are formed. These products are separated and intermediate oil is passed with hydrogen through a relatively high temperature reaction zone (850–1050° F.).

A substantial proportion of the intermediate oil is converted into aromatic hydrocarbons and hydrogen. The hydrogen produced in the vapour phase stage is practically sufficient to run the process and little, if any, need be added. The recirculated gases are preferably purified to remove hydrogen sulphide and gaseous hydrocarbons. An oil wash is usually sufficient for this purpose.

In the liquid phase stage of the process pressures of 50 to 200 atm. may be employed and hydrogen in considerable excess is supplied with suitable catalysts such as chromium or molybdenum oxides. In the vapour phase stage the pressure is above or about 25 atm. The catalysts may be the same as for the high pressure stage. R. P.

1120. Dehydrogenation, Polymerisation and Condensation of Hydrocarbons with the Aid of a Catalyst. Gas Light and Coke Co. and R. H. Griffith. E.P. 349,444, 26.5.31. Appl., 25.1.30.

Oils decompose at temperatures above 400° C. in the presence of a catalyst, with or without added gases. The nature of the change depends on the conditions and may result in the production of large volumes of gases or of cracked or polymerised liquid and solid hydrocarbons. High temperatures and low pressures encourage the former reaction whilst low temperatures and high pressures favour the latter.

Titanium, chromium, and tungsten, or their compounds associated with promotor substances, are very efficient catalysts. For example, titanium compounds in presence of zinc, aluminium, manganese or chromium compounds (1–6 : 100, atomic ratio), or chromium compounds in presence of tin,

boron, cerium or manganese compounds (16–20 : 100, atomic ratio), or tungsten in presence of chromium, molybdenum, uranium, copper, or cadmium compounds (2–7 : 100, atomic ratio).

The promotor substances if employed in other proportions may act as a deterrent upon the catalyst.

Various methods for preparation of the catalysts are specified. As an example to illustrate the invention, paraffin wax m. pt. 60° C. is heated at the boiling point with 5% of a catalyst prepared from zinc acetate and titanium hydroxide. The product had a greatly lowered m. pt., and its aniline point was lowered by 18° after treatment for half an hour. R. P.

1121. Valuable Products from Carbonaceous Materials Consisting of Hydrocarbons of Aromatic Nature. H. O. Elkington, Assr. M. V. de Bataafsche Petr. Mij. E.P. 349,470, 26.5.31. Appl., 26.2.30.

Hydrogenation of aromatic hydrocarbons, *e.g.*, naphthalene and its homologues, is catalysed by molybdenum compounds, particularly the oxide adsorbed by finely-divided coal or carbon. Sulphur, added to the catalytic mass, furthers the hydrogenation. It is claimed that Edeleanu Extract may be so hydrogenated at a temperature of 480° C. H. C. R.

1122. Production of Hydrocarbons Rich in Hydrogen from Tars or the Like. I. G. Farbenind. A.-G. E.P. 349,588, 26.5.31. Appl., 23.1.30.

Hydrocarbons rich in hydrogen may be prepared from tars, petroleum residues, cracked residues, or pitches by treatment in a liquid phase with hydrogen under pressure in the presence of catalysts without the formation of low boiling hydrocarbons. By this process however yields decrease on account of deposits which form on the catalysts and on the inner walls of the apparatus due to the viscous nature of the raw materials.

This may be obviated by dilution of the raw materials with solvents such as tetrahydronaphthalene, decahydronaphthalene, and hydrogenated anthracenes, but hydrocarbons having a similar boiling point range may be employed.

For example, a coal tar of 1·115 density at 20° C. containing 50% of pitch which is practically insoluble in petroleum ether is diluted with 50% of tetrahydronaphthalene. This mixture is hydrogenated at about 420° C. under a pressure of 200 atm. of hydrogen in contact with a catalyst consisting of tungsten sulphide and cobalt oxide. Even after weeks of operation a product is obtained which contains only 5% of pitch which is completely soluble in petroleum ether. Splitting of carbon chains only occurs to a negligible amount and the loss due to the formation of gas is only about 2%.

If the same tar were employed without the diluent the activity of the catalyst decreases after a few days and large deposits of coke are found in the apparatus. R. P.

1123. Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind A.-G. E.P. 349,892, 4.6.31. Appl., 6.3.30.

Alkaline residues from the refining of mineral oils are dehydrated and subjected to a treatment with hydrogen under a pressure of 200 atm. at a temperature of about 435° C. An oil of sp. gr. 0·784 is obtained in a yield of 78% by weight. This oil contains 36% boiling below 180° C.

By subjecting a similar oil to destructive hydrogenation in two stages in the presence of a catalyst prepared from molecular proportions of zinc oxide and molybdic oxide at temperatures of 425 and 455° C. respectively an oil of

0·830 sp. gr. is obtained in 73% yield. This oil contains 24% benzine. Therefore a process is claimed for the manufacture of valuable liquid hydrocarbons, which consist in subjecting residues, obtained in the industrial working up of crude heavy hydrocarbons with alkalies, to a destructive hydrogenation, in the presence or absence of catalysts. R. P.

1124. Conversion of Hydrocarbons Having a High Boiling Point into Hydrocarbons Having a Lower Boiling Point, by Hydrogenation. R. H. Griffith and the Gas Light & Coke Co. E.P. 349,991, 26.5.31. Appl., 25.1.30.

A process of destructive hydrogenation of high-boiling hydrocarbons with the formation of hydrocarbons having a lower b. pt. is described. The use of promoter substances with molybdenum as a catalyst in the hydrogenation has been investigated. If a curve of catalyst-activity be plotted, the abscissa representing the atomic proportion of promoter used relatively to the Mo and the ordinates representing catalyst-activity, such a curve has in it—from say 0% to 10% promoter percentage values—an acute local peak on either side of which the curve is less steep. So a promoter can be chosen by reference to the curves, the selected ratio of promoter to Mo being such that the ratio is within the peak-ratio range. The promoter element is one of the lighter metals, alkaline earth metals, metalloids or solid non-metallic elements. W. D. S.

Chemistry of Petroleum.

1125. Constants of Paraffin Hydrocarbons. C. K. Francis. *Oil and Gas J.*, 1931, **30** (10), 102.

The physical constants of the lighter paraffin hydrocarbons contained in natural gas and in natural gasoline are presented in the form of one table prepared by combining the data obtained by various authorities. The list includes all the normal hydrocarbons from methane to octane and in addition iso-butane and iso-pentane. The properties given include molecular weight, melting and boiling points, heat of vaporisation, gravity in gaseous and liquid form, critical pressures and temperatures, pressure of liquid at various temperatures, coefficients of expansion, specific heat, calorific value of gas and liquid, limits of inflammability, data on flame propagation and various conversions of these constants into values in terms of standard volumes or weights. The sources of information are quoted. R. A. E.

Analysis and Testing.

1126. Gum Testing with the Oxygen Bomb. E. W. Zublin, *Oil and Gas J.*, 1931, **30** (9), 26.

Recent investigations have demonstrated the necessity for a reliable gum test. It is considered that the air jet method devised by Hunn, Fisher and Blackwood gives an accurate and reliable indication of the gum-present in gasoline at the time of test and is well suited for routine work. The copper dish test is unreliable and specifications demanding a low gum-content determined by this method are liable to entail reduction in anti-knock value of the gasoline provided, owing to the treatment necessary.

Some form of oxygen gum test as suggested by Voorhees and Eisenger provides a fairly reliable basis for estimating the potential gum-content of a gasoline, and specifications based on this method have been adopted by the U.S. Army and Navy. The general procedure adopted is described, and improvements in the original method are discussed. It is now thought that an increase in initial oxygen pressure to approx. 100 lb. is preferable and consequently a steel bomb must be employed. Bombs devised by the Army Air Corps and by the Ethyl Gasoline Corp. are described. The dimensions of the former are considered to be more suitable because they permit admission of a larger sample container which reduces possibility of loss of sample by frothing on release of pressure. The closing device adopted by the Air Corps is less satisfactory than that used by the Ethyl Gasoline Corp., and a design giving satisfactory service is described. Accidents which have occurred when using bombs are thought to be due to self-ignition of gasoline owing to high pressure of oxygen admitted consequent upon failure of gauges to register pressure. A safety filling arrangement, employing as an essential part a safety valve permitting a maximum pressure of 150 lb. is described. To secure consistent results the air originally present in the bomb must be displaced by release of, and refilling with, oxygen once or twice.

The test procedure recommended is to fill with oxygen up to a pressure of 120 lb. at 32° F., leave the bomb in an ice bath for 30 to 45 min. until the pressure is constant, indicating absence of leakage. Open valve and reduce pressure to 100 lb., raise bath temperature to 212° F. for duration of test and then chill the bomb quickly in cold water to reduce loss of gasoline by frothing on reducing pressure. For ease of manipulation and to ensure that accurate maintenance of conditions has been obtained, the use of recording gauges is recommended. Recommendations for elimination of leaks in bomb and for the preparation of sample containers are made. A suitable type of water bath is described and illustrated.

Gasolines after exposure to oxygen are often cloudy, due to development of gum insoluble in gasoline. The bottle must therefore be shaken and the sample for test by the air jet method pipetted off as quickly as possible.

The method may be used not only for determining potential gum but also for plant control in treating cracked gasolines. The efficiency of clay when treating vapour-phase gasoline by Gray process may be thus estimated and figures illustrating this point are presented. R. A. E.

1127. Estimation of Water in Alcohol Fuels. K. R. Dietrich & C. Conrad. *Erdöl und Teer*, 1931, **7**, 317-318; *Z. angew. Chem.*, 1931, **44**, 532.

The method described is based on the reaction $Mg_3N_2+6H_2O=3Mg(OH)_2+2NH_3$. At least double the weight of water expected (but not less than 5 gr.) of magnesium nitride is placed in a flask and 50 gr. of the fuel added slowly from a tap-funnel. The liberated ammonia is distilled over into excess of standard sulphuric acid. Excellent results were obtained with ethyl alcohol of from 90·1 to 99·9% concentration. Aluminium and calcium nitrides are not suitable, the former giving low values owing to the reaction being not sufficiently vigorous and the latter giving ethoxides. Ahnydrous methyl alcohol was found to react with magnesium nitride—$Mg_3N_2+6CH_3OH=3Mg(OCH_3)_2+NH_3+N(CH_3)_3$. The occurrence of this reaction is easily detected by the penetrating smell of the trimethylamine. It does not, however, occur unless the concentration of methyl alcohol reaches 60—65%. As alcohol fuels usually contain not more than 10% of methyl

alcohol, good results for water were obtained with this constituent present. If the concentration exceeds 60%, the fuel is diluted with an equal volume of anhydrous ethyl alcohol. E. B. E.

1128. Determination of Small Proportions of Hydrocarbon in Alcohol containing Acetone. R. W. Hoff, *J.S.C.I.*, 1931, **50**, 242-T—244-T.

The method of Babington and Tingle (*J. Ind. Eng. Chem.*, 1919, 11, 555) for the determination of small amounts of benzene or other hydrocarbons volatile in steam in alcohol fails completely when acetone is present.

Most grades of denatured alcohols, legalised by the Canadian Government, which contain hydrocarbons, also contain acetone. The latter is a necessary constituent of the wood alcohol used as a denaturant, and in order to follow the later stages of the above process it is necessary to convert the acetone into a relatively non-volatile derivative. This is done by digesting dilute acetone solutions with aluminium amalgam in the presence of potassium hydroxide, which converts the acetone into a mixture of higher-boiling products, the principal one of which appears to be phorone. The action of the amalgam is obscure. Described briefly the method is as follows:—

After refluxing the alcohol sample with the amalgam and potassium hydroxide (hydrogen being slowly evolved), water is added and the mixture distilled. The first two 20 ml. distillates are collected separately in Eggertz tubes and treated with potassium dichromate and hydrochloric acid. A known volume of low boiling petroleum spirit is added and, after well mixing, from the volume of the hydrocarbon layer the proportion of hydrocarbon in the sample is obtained.

A table is given of results obtained on mixtures of known compositions.

The continuous evolution of hydrogen is said to cause loss of volatile hydrocarbons such as benzene, so a different method of "fixing" the acetone was introduced, depending on its reaction with hydroxylamine in the presence of alkali.

The distillate, after removal of any pine oil present, is treated with a slight excess of hydroxylamine and made alkaline to phenolphthalein with potassium hydroxide. After diluting, the mixture is distilled and the hydrocarbons estimated in the first portions of the distillate as above.

A table is given showing the results obtained on mixtures of known composition. W. A. W.

1129. Estimation of Solid Foreign Matter in Lubricating Oils. H. Burstin. *Erdöl und Teer*, 1931, 7, 335–336.

The author criticises the method for estimating solid matter in lubricating oils which has been adopted by the German Society for Testing Materials The quantity of oil (5–10 grs.) recommended to be taken for the determination is too small, as the weight of residue on the filter is often only ½ or 1 mg. The variation in weight of the filter-paper and weighing bottle due to temperature and atmospheric changes may approach this weight. Results by this method may vary by as much as 100%. The author recommends using 10–100 grs. of material in 50–200 c.c. pure benzene, the filter being previously degreased by washing with benzol and drying at 105° C.

The solid matter usually present consists of colloidal coke, colouring matter, dust, rust or glue from barrels. The limit given in the specification is 0.01%. This limit should be different for various classes of oil; *e.g.*, greater for heavy cylinder oils than for transformer oils. E. B. E.

1130. Carbonisation of Lubricating Oils. H. N. Bassett. *J.S.C.I.*, 1931, **50**, 527–529.

The various tests usually employed to determine coke formation are mentioned, and very brief summaries are given of the published work of several investigators.

The author concludes that no satisfactory test has yet been devised for oxidising a lubricating oil under conditions comparable with those existing in an automobile engine. W. A. W.

1131. Calculation of Critical Viscosity and the Validity of Stokes' Law in Tausz' Falling Sphere Viscometer. A. Rabl and F. Geiger. *Petr. Zeit.*, 1931, **27**, 439–442.

In this work Arnold's equations for spheres falling into a viscous liquid are applied to Tausz' falling sphere viscometer. This represents a system in which a sphere falls into a cylinder of very small radius. The expression $r_c \rho \nu / 0.6 = 1$, ($r < 0.6\, r_c$) is derived from Arnold's equations, and the extreme limit of the validity of Stoke's law is defined by the equation $r \rho \nu / \eta = 0.6$; r represents the radius of the sphere, ρ the specific gravity of the sphere, ν the velocity with which the sphere falls, η the viscosity, r_c, η_c the critical radius and critical viscosity respectively. Reynold's criteria for a capillary system and a concentrated cylinder are analogous.

The interpretation of critical viscosity is discussed and is defined as that viscosity at which the stream-line flow in a system changes to turbulent flow. Rabl and Geiger calculated critical viscosity from the equation $\eta_c^2 = r \rho s c / 0.6$ where s represents the distance through which the sphere falls, and c is a multiplication factor.

It was shown that approximate linear relations existed between the critical viscosity and density of the sphere on the one side and the critical viscosity and the multiplication factor on the other side when r and s were constant; η_c and c can then be evaluated.

Stoke's law is valid for Tausz' apparatus provided it is multiplied by a correction factor A where $A = 9sc/2r^2\ (\rho - \rho^1)\rho$, ρ^1 representing the specific gravity of the liquid, $g = 980.6$ cm./sec.^{2}. The multiplication factor c was found to be proportional to the difference between the specific gravity of the ball and of the liquid so that $c = k\ (\rho - \rho^1)$.

When Tausz' viscometer is used with a steel sphere c (= 45) and the lower limits of application are known; then $\eta_c = 21.48$ centipoises (3.3° E). When spheres of equal radii, but different specific gravity are used c and the lower limits of application need only be determined. The multiplication factor can be found by calculation from the equation $c = k(\rho - \rho^1)$, or by graphical interpolation. The lower limits of application can be determined from the formula for η_c, or similarly by graphical interpolation; η_c for the two glass spheres utilised in this work is found to be 5.87 centipoises (1.5° E) and 11.96 centipoises (2.2° E) respectively. W. S. E. C.

1132. Laboratory Determination of Sulphur Compounds in Petroleum Gases and Liquids. A. W. Trusty. *Refiner*, 1931, **10** (7), 91.

Hydrogen sulphide in natural gas is determined by absorption in standard iodine solution, but owing to the presence of unsaturated compounds in refinery gases this method is not applicable to the determination of sulphur in such samples. Absorption in lead nitrate with estimation of unspent lead nitrate by means of ammonium molybdate is recommended. For the estimation of sulphur compounds in light oils the method of Faragher and Morrell (*Ind. Eng. Chem.*, Nov., 1927) is suggested and briefly outlined. W. D. S.

1133. Determination of the Percentage of Bitumen in Asphaltic Materials. D. M. Wilson. *J.S.C.I.*, 1931, 50, 599–600.

A method is described which claims to have overcome the difficulty of obtaining a true figure for the content of Asphaltic Bitumen soluble in carbon disulphide, in a mixture of bitumen with mineral aggregate.

Since the proportion of bitumen is usually determined by difference, any loss of "filler" by creepage, aided by the extremely volatile solvent, would be reported as bitumen with a corresponding serious error in the result. This source of error has been overcome by placing the filter papers in a large funnel, the top edge of which is ground flat, and covering the funnel with a glass plate with a small hole in the centre. The stem of a small funnel is placed through this hole to dip just below the surface of the liquid in the large funnel. Solvent is introduced through the small funnel.

An alternative method for mastic mixtures is also given, wherein a closed separating funnel is used in place of the small funnel to hold the solvent; and by adjustment of the position of this separating funnel, the addition of the solvent can be automatic. This also obviates the possibility of disturbing the aggregate which would tend to clog the filter paper. Replacement of carbon disulphide by non-inflammable trichlorethylene is suggested.

The usual details of operation are also given. W. A. W.

Anti-Detonation.

1134. Co-Operative Fuel Research Committee Engine. H. L. Horning. *J. S. A. E.*, 1931, 28, 637–641.

The requirements of a universal fuel-testing engine are outlined, and a description of the present C.F.R. Committee engine is given with diagrams and specifications. The engine can be converted to either a fixed-compression L-head type or a variable compression O.H.V. type, and is suitable for throttle, spark, bouncing-pin, listening-post, or variable compression method of testing. The design is very rugged and the cost comparatively low.

Special features include the valve-rocker-arm linkage, which automatically maintains a uniform tappet clearance as the cylinder is raised or lowered, and varies the compression-ratio.

The new variable compression head is one-piece in order to eliminate gasket troubles and cooling difficulties.

The crankshaft is exceptionally heavy to enable the engine to run for long periods under detonating conditions. The bore and stroke are 3¼in. × 4½ in., which corresponds to the average passenger car engine. Magneto ignition is now used.

The engine has been used for some time by Committee members for research and control work. E. B. E.

1135. High-Temperature Knock Testing. G. Edgar. *Aircraft Eng.*, 1931, 3, 171.

Results are given of a co-operative programme (in which six American laboratories took part) undertaken to obtain information as to the precision of Knock Testing with a high jacket temperature, under the conditions of the U.S. Army Air Force Specification. Five fuels were tested in the S. 30 engine under specified conditions and determinations were made in each case of the octane number of the fuel and the amount of tetraethyl lead necessary to make each fuel equal to 87 octane.

Experimental data are given in a table while a series of curves shows the relation between tetraethyl lead content in ccs. plotted against octane numbers.

For straight-run fuels and benzol blends, the average deviation is about ½ an octane number, and the maximum deviation about 1 octane number. With 100% cracked stock, the agreement is not so good.

Additional data obtained showed that advancing the spark from 14 to 22 deg. gave results essentially the same. Results obtained with the temperature plug as well as the bouncing pin are of the same order at 600 r.p.m., but do not always agree at 900 r.p.m. C. C.

1136. Peroxidation of Hydrocarbons during Combustion in Air. E. Mardles. *Nature*, 1931, 128, 116.

The author points out that the presence of methyl alcohol in the oxidation products of methane can be explained by the peroxide theory, as well as by the hydroxylation theory (*Abst.* 659). Alkyl hydrogen peroxides might be formed by the inclusion of the oxygen molecule in a CH grouping and molecular disruption of the primary peroxide gives alcohols, esters, glycols, etc. It has been shown that methyl hydrogen peroxide can give methyl alcohol on decomposition, the peroxides being formed primarily from ethane during slow combustion. Methane has a low temperature coefficient of gaseous reaction which suggests that autoxidation by the moloxide occurs to an important extent. C. C.

1137. Investigation of Infra Red Radiation from an Engine. S. Steele. *Nature*, 1931, 128, 185.

The infra-red radiation from a single-cylinder Otto cycle engine delivering power has been investigated, by a small fluorite window, for which 31 positions, distributed symmetrically over the combustion chamber, are available. By mirrors, the radiation is brought to a focus on the slit of a stroboscope, which can be adjusted to remain open for any period up to 20° of crank angle at any point of the cycle. The divergent beam beyond the stroboscope is reflected from a mirror and focussed on a sensitive single function Sb-Bi vacuum thermocouple, the E.M.F. being measured on a Moll galvanometer. Curves are given (plotted on a crank angle base) showing (1) radiation to 4.4μ, (2) radiation to 10μ, (3) pressure in the combustion space; the engine was operating without detonation at 900 r.p.m. on commercial petrol S.G. 0.733.

Liquid or gaseous fuels can be used and information on combustion, depth of reaction zone behind the flame front and detonation are to be sought. C. C.

See also Abstract No. 1865.

Engines.

1138. Oberhaensli 4-Cylinder Automotive-Type Diesel Engine. K. Neumann. *Mech. Eng.*, 1931, **53**, 533–534; *Z. Ver. deuts.*, 1931, **75**, 453–455.

This is a single-acting four-stroke cycle engine using Bosch injection valve for fuel injection with a Bosch or Ref pump.

The combustion chamber is separated from the cylinder by a throat, and has inside it a spherical bowl so arranged that the air in process of compression coming from the cylinder is divided into two streams, both characterized by a high degree of turbulence.

So far, the engines built have a nominal output of 80 h.p. at 1350 r.p.m., bore of 130 mm. (5.1 in.) and stroke of 180 mm. (7.1 in.). The piston velocity is 8.1 m. (26.57 ft.) per sec. The specific fuel consumption is claimed to remain constant over a considerable range of load, and when the output of the dynamo and fan is subtracted, is about 200 gr. per h.p. hour (0.44 lb. per h.p. hour). At maximum output the engine delivered 102.8 h.p. with a mean piston pressure of 7.02 atm. W. A. W.

1139. Status of Compression-Ignition Engine. E. T. Vincent. *J.S.A.E.*, 1931, **28**, 642–647.

After a short review of the development of the subject, the author describes modern views on spray design, injection, turbulence and combustion chamber design.

The advantage of turbulence depends very largely on the jet used. Detonation is comparatively harmless in compression-ignition engines, the chief objection to it being the noise and rapid rate of loading the bearings. Increasing compression-pressure eliminates detonation in this type of engine.

The author considers that the compression-ignition engine of the future will embody a direct injection fuel-system. The weight of the engine should become not more than 10 to 15 per cent. greater than that of the corresponding gasoline engine and may even become less than that of the present gasoline engine. E. B. E.

1140. Cooling of Aero Engines (Experiments Undertaken to Show the Possibilities of High Temperature Liquid Cooling). A. Nutt. *Aircraft Eng.*, 1931, **3**, 172–174.

Increasing the temperature of the coolant gives a saving in weight, a higher air speed by reduction of drag, the possibility of decreasing fuel consumption and simplified controls. Satisfactory liquids from Freezing Pt., Viscosity, and B.P. standards, are ethylene glycol and diethylene glycol, particularly the latter mixed with 5 per cent. of water. The necessary adjustments of clearances, etc., must be made for an engine to work at the higher temperatures. An increased supply of oil is required and the latter must be suitably cooled. Great care must be taken with all joints, special gaskets and special rubber connections being required.

Tests have been carried out to evaluate high temperature cooling on a 600 h.p. liquid cooled Conqueror engine using Californian petrol of Octane No. 73. Sufficient tetraethyl lead was added under each condition entirely to suppress detonation. Tests were made at three outlet temperatures, 180° F., 240° F. and 300° F., and at three compression ratios, 6.1, 7.22 and 7.8 to 1. There is a loss of power as the temperature rises, but the specific fuel consumption improves (at full throttle) up to 240° F. but at 300° F. it is no better than at 180° F. At the higher compression ratios there is an improvement in specific fuel consumption. The effect of changing compression ratio on the h.p. developed at different outlet coolant temperatures was studied. The power loss necessary to obtain best economy is less at the higher temperatures and with the higher compression ratios, while 10 per cent. of fuel can be saved by raising the compression ratio from 6.1 to 7.8 to 1.

Curves are given illustrating the above points.

The importance of fuel of good anti-knock quality is emphasised. H.P. Curves are also given showing the result of changing the compression ratio and the outlet coolant temperature, the b.m.e.p. being also indicated.

It is considered that 240–260° F. is a very satisfactory operating temperature.

A final series of curves shows the best economy conditions under various conditions of temperature and compression ratio. C. C.

1141. Charge Mixing Device for Internal Combustion Engines. R. Mollart. E.P. 344, 985, 19.3.31. Appl., 4.1.30.

The apparatus consists of a hollow cone, through which a number of holes are drilled tangentially, set base to base with a hollow truncated conical body. The mixer is placed in the induction pipe of the engine, with the apex of the drilled cone towards the carburettor. The gases are separated into streams and each stream given a rotary motion by its passage through the holes. A further mixing and hence a richer mixture is obtained by an added swirling motion due to the holes being drilled tangentially to the cones surface.

J. M. F.

1142. Ensuring the Ignition in Spontaneously Igniting Engines. W. W. Triggs. E.P. 345,024, 19.3.31. Appl., 29.1.30.

The invention provides an arrangement for ensuring ignition when starting spontaneous ignition engines particularly of the Diesel type having a pre-combustion chamber. This is effected by pumping a readily ignitable fuel in a finely divided state into the air inlet pipe.

Thus the ignition of the heavy fuel, injected simultaneously in the normal way, will be ensured without altering the compression pressure by the spontaneous ignition of the readily ignitable fuel.

J. M. F.

1143. Fuel Pumps for Oil Engines. Swiss Locomotive and Machine Works. E.P. 345,134–5, 19.3.31. Appl., 24.4.30.

The invention provides a fuel pump for oil or Diesel engines in which a yieldable receiver of relatively small capacity communicates with the pump delivery, and is situated within the pump assembly between the pump chamber and the delivery valve. This vessel may be arranged so as to receive fuel only up to a given maximum pressure in the pump, and so that its action may be changed or entirely suspended.

With this arrangement the rise and fall in pressure is relatively slow, and so shock to the pump valves and injection apparatus is obviated. The automatic fuel valves in the combustion cylinders do not operate suddenly, thus preventing large increases in pressure and oscillations of these valves, which are highly injurious to the process of injection and to the combustion of the fuel.

J. M. F.

General.

1144. New Type Tank Car Adopted by Empire for Shipping Butane, *Oil and Gas J.*, 1931, **30** (10), 90.

Five tank cars for the transportation of liquified commercial butane built to I.C.C. Specification 104A have recently been placed in operation. The cars have a shell capacity of 11,000 gal., but have no expansion dome. They are loaded with 10,200/10,300 gal. at 60° F.

The cars have no bottom connections, so that unloading by gravity is precluded. All the connections, gauging apparatus, etc., are situated within a steel housing appearing on top of the car. Within this housing are (1) two valves connected to pipes running to the bottom of the car for filling and emptying contents; (2) Connection to the vapour space to control pressures, and provision of a safety valve set for a maximum pressure of 75 lb.; (3) a sliding tube gauge and a thermometer well for measuring purposes.

Steam coils are placed against the shell of the car near the bottom so as to increase the pressure of the contents, if necessary, for unloading. The shell is covered with 4 in., and the steam coils with 2 in., of cork insulation protected by a ¼th in. steel jacket. The tanks are 9/16th in. in thickness and must be able to withstand a hydraulic pressure of 100 lb./sq. in.

R. A. E.

ABSTRACTS.

Geology.

1145. Problem of Dry or Unsaturated Strata. J. Versluys. *Petr. Times*, 1931, **26**, 193–195; *Proc. K. Akad, Wetensch, Amsterdam*, 1931, **34**, 591–597.

The existence of unsaturated strata at depth, overlain by water-filled beds, is considered very improbable. Such a hypothesis has only been put forward to connect synclinal oil occurrence with the buoyancy theory.

In the author's opinion this phenomenon can be explained on the assumption that formations containing oil outside the anticlines have variable porosities. The anticlinal and synclinal accumulation of oil and gas would therefore appear to be due to water flowage and not to buoyancy. Moreover, having once penetrated the coarser deposits the tendency would be for the oil to remain in them. P. A. S.

1146. Exceptional Association of Oil and Water in Producing Zones at Refugio, Texas. R. G. Maxwell. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 953–964.

The Refugio field occupies an area of approximately 4½ square miles. The surface beds are considered to be Lissie and younger in age, the oldest formation so far penetrated being Upper Jackson. Production has been from the Catahoula-Gueydan and older formations, and the oil-bearing sand horizons from 3600 ft. to 6400 ft. are briefly discussed.

Salt water, which is of common occurrence above oil and gas in the field, has resulted in erratic production and consequent high development costs; many wells have had to be drilled rather than cored through the beds.

Variable porosity and the overlapping of sand lenses are thought to be responsible for this exceptional association of oil and water. The author considers such conditions are probably common to all Gulf Coastal fields where lenticular sands predominate. G. S. S.

1147. History of Development and Geology of East Texas Fields. L. G. E. Bignell. *Oil and Gas J.*, 1931, **30** (13), 25.

The East Texas Fields cover a large portion of the western halves of Rusk and Gregg Cos., extending over to the west from Rusk to Smith Co., and into Upshur Co. which adjoins Gregg. It is approximately 40 miles in length with a maximum width at the northern end of about 7½ miles.

The discovery well or joiner pool was located at the eastern edge of the pool. The field, now 11 months old, is defined along the western edge, though open territory still remains to the north and south which may be productive. From present indications it is estimated that the pool will contain 90,000 acres in one continuous field.

It is generally agreed that the formation from which the oil originates is Woodbine in character. The producing sands vary greatly in porosity; it is stated this ranges from 10 to 27%. Tight sands below 15 or 16%, however, are not productive of sufficient oil to make good commercial wells. This range in porosity is due to some degree to the deposition of a volcanic ash (bentonite).

An estimate of the total production is given as 1,350,000,000 brls.: 90,000 acres at 15,000 brls. per acre. Factors largely depending on such recovery, however, will include location of wells and their method of operation, porosity and water movement through the sands. G. S. S.

1148. Salt Water Encroachment in East Texas. J. S. Hudnall. *Oil and Gas J.*, 1931, **30** (16), 61.

The author claims that danger from salt water intrusion in the East Texas pool is not alarming or threatening. Considering the development features, and the amount of oil that has been produced, the salt water problem is small.

Water encroachment is now limited to wells drilled to a depth of 3310–3320 ft. sub-sea level. Theoretically, the water table rises 1 ft. for every 30,000,000 brls. of oil extracted; oil produced to date (56,000,000 brls.) would raise the table about 2 ft. Thus to prevent water trouble, drilling should not be attempted at a greater depth than 3290 ft. sub-sea. A map showing the salt water encroachment is included. G. S. S.

1149. Geology and Economic Importance of the East Texas Field. J. S. Hudnall. *Oil Weekly*, 1931, **62** (7), 43.

Accumulation occurs on the top or eastern limit of the Woodbine Sand which dips west 40 ft. to the mile. The sand, Eagleford in age, is deposited on the flank of the Sabine Uplift, the dip being due to progressive elevation along its axes. It is overlain by Eagleford shale on the west and Austin chalk on the east. The deposit is about 50 ft. thick over the area, with 30 ft. of saturation and 20% porosity. The water encroachment is normal, the water table underlying the oil on the western side.

The maximum life of the pool is estimated at 10 years. Included in the paper are geological maps and sections and production data. P. A. S.

1150. East Texas Salt Domes and Structures Promise Oil. J. Logan. *Oil Weekly*, 1931, **62** (8), 17.

Much speculation exists as to whether the interior salt domes and structures occurring in East Texas will eventually become important sources of oil.

The domes occur in the middle of the East Texas Basin, in which region great thicknesses of Woodbine Sand are present. Up through this and overlying formations the domes have been thrust, and usually result in the strata being tilted around them. Moreover, on some of the deeper domes, sands have been raised above them without being pierced; in such cases structural conditions are extremely favourable for oil accumulation. The presence of faulting around the domes is also said to be of much importance in this connexion.

In addition to the Woodbine Sand, other oil-bearing formations above and below it are found in the East Texas Basin. Petroleum possibilities of the domes, therefore, extend also to those formations and accordingly are briefly reviewed.

Accompanying the paper is a structural map and a table giving details of the domes and related structures in East Texas. G. S. S.

1151. Oklahoma Deep Test Reveals Valuable Geologic Data. F. Swindell. *Oil Weekly*, 1931, **62** (8), 14.

A record drilling depth of 8,662 ft. was recently established in the Mid-Continent area at Cement Pool, Oklahoma. As a result new and important geological data have been obtained. Hitherto, knowledge of the geology of this part of the State had been limited almost entirely to that acquired by study of the surface rocks.

A partial log is given showing a thickness of 7225 ft. of the Pennsylvanian formation has so far been penetrated, a thickness much greater than was formerly thought to be present. Opinions differ considerably, however, regarding the section of the Pennsylvanian represented. Some geologists

believe the Central Oklahoma or Oklahoma City type occurs, while others consider the typical Southern Oklahoma section is being encountered. Hard sands and shales occurring at about 8300 ft. have been correlated as Middle Deese.

Productive oil sands in certain fields of Carter Co. have been correlated with the Dornick Hills formation which underlies the Deese. It is possible, therefore, that the beds in which oil and gas was encountered at 8395–8419 ft. are equivalent to the Dornick Hills. A major unconformity is expected to be met at the base of the Pennsylvanian Series.

Included in the paper is a geological section extending from Carson to Cleveland counties. G. S. S.

1152. General Geology of North-East Mexico. J. L. Tatum. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 867–893.

In this paper descriptions are given of the Tertiary formations with their distribution in North-East Mexico, and correlation made with equivalents in Texas. Brief reference is made to the Cretaceous rocks in describing the changes from the Texas to the Mexican section, and to their probable contribution as source material for the younger sediments.

The Mexican Eocene differs from that of Texas only in the fact that it is somewhat thicker and is practically all marine. It consists of the Wilcox, Claiborne and Jackson Groups. Unconformably overlying the latter is a thick series of Oligocene deposits (mainly volcanic), and similar to those of Gueydan in S. Texas. These are followed by sandstones, interbedded with clays, of Miocene age and younger.

Mention is made of the known structures and trends, together with their probable origin and age of uplift.

The author concludes that North-East Mexico is likely to be an important oil-producing region. It is probably more favourable than South-West Texas owing to the Eocene and younger beds being more marine, and to the presence of many large and well defined structures. G. S. S.

1153. Venezuelan Oil-Field Waters. J. E. Smith. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 895–909.

Attention is drawn to the presence of naphthenic acids in Venezuelan oilfield waters and a method devised for the measurement of such acids is given in detail. Maracaibo Basin waters are differentiated and classified by the sulphate-carbonate ratio, and many type analyses are recorded in this connection.

It is shown that formations ranging from Cretaceous to Oligocene in the Maracaibo Basin were deposited in water of marine type. The Icotea beds of the Bolivar district may be considered terrestrial in origin from the top of the Oligocene to the basal Miocene. Definitely marine Miocene beds follow in which connate water would be expected, and above these occur the Maracaibo series (younger Tertiary).

It is probable therefore that saline or at least brackish water will be present in all the pre-Miocene deposits, and in only a small part of the post-Oligocene are positively saline connate waters likely to occur.

Outside the Maracaibo Basin proper (e.g., the Mamon and Rio Seco wells), a saline connate water has been proved in the Middle and Upper Miocene, but here geological conditions are different. G. S. S.

1154. Deep Sand Development in Tioga County, Pennsylvania. J. Gaddess. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 925–937.

The Tioga Co. gas field is in the eastern part of Farmington Township and occupies an area 7 miles long and 3 miles wide, or approximately 7000 acres.

The surface structure is an elongate dome on the Sabinsville anticline, the rocks being of Upper Devonian age (Chemung formation) and consisting of thinly bedded sandstones, shales and limestones. Underlying these beds are the Portage, Genesee and Hamilton formations, black bituminous shales of the Marcellus and the Onondaga limestone. The dip of the formations increases with depth, particularly so on the south flank of the dome, and final development may show 500 ft. of closure on the Oriskany sand.

In all wells drilled to date the formations penetrated have been uniform down to the Onondaga limestone. Here, however, a fault is proved having an estimated throw of at least 350 ft., but further drilling is necessary before its extent can be determined. The presence of this fault is a matter of primary interest in the field, as it appears to be a controlling factor in the formation of the fold and the accumulation of gas.

Development has been rapid; early in 1931 there were eight completed wells on the Farmington structure, four of which were producing gas wells with a flow of approximately 120,000,000 cu. ft. per day. G. S. S.

1155. Richland Gas Field, Richland Parish, Louisiana. D. Gordon. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 939–952.

The Richland Gas Field is situated in North-Eastern Louisiana in the Mississippi embayment; it lies 45 miles west of Mississippi River and 10 miles south-east of the Monroe gas field.

The following formations occur in the area: Quaternary, Eocene (Claiborne, Wilcox, Midway Clays), and Upper and Lower Cretaceous. A generalised section of these is given, together with brief descriptions of each.

The principal producing horizon occurs in the lower part of the Upper Cretaceous (Tokio formation) at depths ranging from 2320 ft. to 2500 ft. over an area of more than 75 sq. miles. The deposits consist of porous tufaceous sands and red clay.

Tectonic features of the field have been determined from well records on the base of the Midway clays; this is equivalent to the top of the Tokio in practically the whole producing area. The structure is an anticlinal fold with a dip of 80 ft. to the mile on the south-east side of the field and lesser dips on the other sides; only a low structural saddle separates this field from the Monroe.

The drilling method employed was to set 6-in. casing at 2300 ft., and then drill to 2450 ft. and bring in the well; the wells are completed with or without tubing. At present, practically all of them produce through tubing, the advantages being that the wells can meet increased demand for gas and, if water is drawn into them, it can be handled by the tubing acting as a syphon.

The production from 194 wells by January, 1931, amounted to 141,000,000,000 cu. ft. of metered gas.

The paper is accompanied by three maps. G. S. S.

Field Technology.

1156. Protective Coatings for Pipelines. *Chem. Met. Eng.*, 1931, **38**, 402.

Tests carried out in the laboratories of the Institute of Paint and Varnish Research indicated that the present steel pipelines give the best all-round satisfaction, but that they needed protective coatings.

The criteria for ideal protective coatings are given as :—Cheapness ; ease of application ; resistance to soil waters and to deformation by contact with soil ; non-conducting in nature ; adhesion during settling of soil or expansion of pipe ; stability to temperature changes ; adhesion to standard top coats ; resistance to shock during back filling ; adaptability to repairs and resistance to acids, alkalies and petroleum oils.

The most satisfactory coatings were found to be : (*a*) zinc chromate primer coated with viscose, pyroxylin, varnish, rubber tape or aluminium foil ; and (*b*) metal casing jointed to the pipe with sulphur, zinc dust, paraffin, diphenyl, resin cement, rubber or resin.

Tests on a series of semi-permanent transparent coatings indicated that raw oil paints were too slow drying but had high durability. Heat-treated oils containing 10% of zinc chromate were found the most satisfactory, while asphalt varnishes failed on exposure to the sun. C. L. G.

1157. Cleaning Out Gas Wells Under Pressure by the Tubing Method. J. S. Ross. *Int. Petr. Techn.*, 1931, **8**, 403.

The method developed by the Southern States Co. for the cleaning out of gas wells has met with remarkable success, and has proved itself to be safer, quicker and more economical than the Cable tool system formerly used.

The wells were cleaned under pressure through tubing, and the waste experienced when the wells were opened to atmosphere was eliminated.

In the course of experimental work it was found that the caving material met with consisted of bentonitic shale, which readily absorbs water and rapidly disintegrates. When thoroughly mixed with water it can be syphoned out by natural well pressure.

The equipment used consisted of the regular snubbing outfit used for running 2 in. tubing against pressures of about 400 lbs., a special tubing swivel, a high pressure hose and a water pump, capable of delivering a pressure of about 1000 lbs. The hose was connected between the swivel and a 2 in. rise leading to the water pump with a side connection to the casing head.

To the lower end of the tubing, blades were welded, and a cast iron disc was inserted in the collar above this joint. The tubing pressure was first equalised with the casing pressure, and the disc was broken by a sharp pointed punch, lowered on the end of a measuring line through a stuffing box and lubricator at the surface.

With the tubing open to the atmosphere, the string was slowly lowered and the water and mud syphoned out. The tubing swivel was then screwed to the tubing and connected to the pump, with the valve "A" at the top of the swivel closed and that on the hose connection "B" opened ; water was pumped down the tubing as it was rotated and lowered into the cavings. The tubing was then picked up a few feet, the valve on the pump line "E" was closed, and the by-pass valve "D" on the flow head line was opened. When the pressure in the tubing was equalised, valve "B" was closed and valve "A" opened, and the freshly mixed mud syphoned out while the tubing was lowered and rotated.

This procedure was repeated many times. The tubing was then pulled up and suspended on a tubing packing head. L. V. W. C.

1158. Bottom-Hole Beaning has Useful Application on Producing Gas Wells. K. C. Sclater. *Petr. Eng.*, 1931, **2** (11), 23.

Bottom-hole beaning has resulted in a more efficient operation of producing gas wells. The remarkable type of choke in the Louisiana high pressure gas fields has an important application in the better operation of gas wells.

Where water is present in the well, a bottom-hole choke reduces the well delivery pressure to the point where the hazard of freezing of the well connections is eliminated and also, when inserted in the well syphon, aids in the removal of water with a minimum loss of gas.

The freezing at high pressure causes a great deal of trouble, and the bottom-hole choke would seem to offer a satisfactory solution to this problem. By the installation of a removable choke nipple in the lower end of the tubing of a high pressure gas well, it is possible to operate during the coldest periods of the year with a minimum of trouble and at a high efficiency. Providing the size of the choke opening is properly designed, the well will produce with a minimum pressure drop, and no freezing will be experienced at the surface, as the gas, after passing through the choke, will expand gradually. Thus the chilling effect produced by the expanding gas will be dissipated.

An actual test has shown that it is possible to reduce the well casinghead pressure to only 7½ lb. sq. in. with a bottom-hole choke, whilst with a surface choke the well showed a casinghead pressure of 1200 lb. sq. in. Further tests have shown that by increasing the size of the choke, casinghead pressures up to 1000 lb. sq. in. can be obtained, thus demonstrating the wide range of pressures and well conditions under which bottom-hole chokes can be usefully employed.

In the Mid-Continent it is the practice to produce a gas well through 2 in. tubing which also serves as a syphon for the removal of any water that may be present. As the volume and pressure of the gas diminish, the velocity of the gas is correspondingly lowered, and eventually is not great enough to carry off the water as fast as it is made. When this occurs, it is customary to open up the syphon at intervals and blow off the accumulated water. As the water accumulates the well will not produce against the line pressure in sufficient volume, and the blowing-off process has to be repeated. This is a costly process because of the excessive waste of gas it entails.

Eventually a stage is reached when the syphon must be left open continuously to the atmosphere in order to keep the well producing at a reasonable rate.

Again this intermittent blowing off the syphon is liable to damage the well if the sand is loose or tends to cave. This latter condition led to the making of tests with bottom-hole chokes, and the development of these chokes marks an important and progressive step in the operation of gas wells. L. V. W. C.

Crude Oil.

1159. Treating Emulsions on Leases of Small Production. R. T. Bright. *Petr. Eng.*, 1931, **2** (11), 75.

A description of an efficient plant for treating emulsions with a capacity of 80-100 brls./day is given. The lead lines from the wells giving cut oil are brought together to a manifold where the chemicals are introduced. The emulsion and chemicals are mixed by agitation in the lead line and pass to the combination conductor pipe and gas trap. The conductor pipe is best placed over the wash tank, the gas being drawn off for supply to the heater, etc. Experience has shown that the oil emulsion should pass into the wash tank about 18 in. from the bottom. The water in the tank is kept warm (about 120° F.) by circulation through the heater, which can be made from a joint of old casing. The water level in the tank should be not less than 3 or 4 ft., being maintained at this level automatically by means of a syphon. In many cases a better wash and a longer settling out time can be maintained by increasing the amount of water in the tank to about one half of the height of the tank. C. L. G.

1160. Egyptian Crude Oils Vary Widely. E. Egloff, E. F. Nelson, P. Truesdell. *Oil & Gas. J.*, 1931, **30** (12), 22.

The crude oil from the three fields in Egypt, Hurghada, Gemsah, and Abu Durba, differs considerably in character. Hurghada crude, found at depth from 1600 ft. to 2000 ft., is a pleasant smelling, heavy, brown black oil of mixed asphaltic and paraffinic character. Gemsah crude is a light, paraffin base oil of a greenish black colour, produced from 1290 ft., while Abu Durba is a heavy asphaltic crude thought to be an oxygenated residual product of petroleum.

The properties of these crudes are given in a table, it being shown that Hurghada crude yields 8% gasoline and 15% kerosine, while Gemsah crude yields 27% gasoline and 32.4% kerosine. Abu Durba crude yields 20% benzine and kerosine. The production of crude oil in Egypt is given as 1,911,000 brls. in 1929.

There are two refineries in Egypt, that of the Anglo-Egyptian Oilfields with a capacity of 1500 tons daily, and that of the Egyptian Government with a capacity of 60 tons daily, both at Suez; 5% of the domestic production in Egypt goes to the Government as royalty, and is worked up at their refinery, together with a certain amount of purchased oils.

In the Anglo-Egyptian Refinery distillates from the asphalt plant are cracked in the Dubbs plant, together with press. dist. bottoms and Egyptian solar oils. The flashed residuum method of operation is used, one particular run lasting 47 days and processing 113,518 brls. of oil yielding 55% gasoline.

Details of various cracking runs giving operating conditions, yields and properties of products are given. Lime is injected during cracking at the rate of ½ lb./brl. charging stock to minimise corrosion. The press. dist. is treated with plumbite and acid and redistilled, giving a product with a I.B.P. of 100° F. and end point of 419° F. It is water white, stable, of good knock rating, and is marketed in Egypt. The residue is a fuel oil of visc. 99–486 secs. Saybolt Univ. at 212° F., with B. S. & W. of 0.8%. C. L. G.

1161. Breaking Petroleum Emulsions. M. de Groote, L. T. Monson and E. H. Keiser, Assrs. to Tretolite Co. U.S.P. 1,812,393, 30.6.31. Appl., 3.3.30.

Emulsions are subjected to the action of a demulsifying agent containing a water soluble ammonium salt of an acetylated sulphur containing derivative of a hydroxylated fatty acid (castor oil) e.g., acetyl-sulphoricinoleic acid. W. H. T.

See also Abstract No. 1239.

Gas.

1162. Developing Markets for Cracked Gas. A. Fisher. *Oil and Gas J.*, 1931, **30** (14), 24.

The sale of gases from cracking stills has been facilitated by recent developments in gas manufacture and distribution. Some are sold as raw material for the manufacture of synthetic products, but at present the most fruitful field lies in converting the gases for distribution to domestic consumers to supplement either natural or manufactured gases. The possibilities are indicated by the increase in consumption of manufactured gas in the U.S.A. from 112,500 mil. cu. ft. in 1905 to 535,000 mil. cu. ft. in 1929, and the consumption of nearly 2 billion cu. ft. natural gas in 1929. The difficulties in negotiating with gas companies are: (1) gases must be brought within certain specifications entailing increased equipment; (2) fluctuating supply according to refinery operations. In the manufactured gas field the scope

as cold enrichment gas is limited owing to gravity limitations and competition with fuel oil now used for enrichment. Two methods of "reforming" the cracked gases to meet commercial needs have been devised. The first consists of first removing sulphur, if necessary, and then passing the gases through the generator beds of standard water gas sets with steam and reducing the gravity and calorific value by cracking. It has been found economical to produce gas directly from the generators at a lower calorific value than actually required, and to enrich with untreated refinery gases to the required standard. This method is particularly suitable for producing material to supplement manufactured gas supplies, and one volume of still gases yields 2½ to 3 volumes of the lower B.Th.U. gas. The second method, which is more applicable to supplementing natural gas supplies than manufactured gas on account of the effect of nitrogen content on the gravity of the product, consists of leading pre-heated air and gas to be "reformed" into a refractory lined steel shell. The gas is partly burned, and the degree of combustion controls the calorific value of the final product. Analyses and calorific values of refinery gas and of the "reformed" gases obtained by the two methods of operation are tabulated, and the possibilities and suitable application of the process discussed. R. A. E.

1163. Treatment of Gases Containing Hydrocarbons. T. S. Wheeler, W. Francis and I.C.I., Ltd. E.P. 351,994, 29.6.31. Appl., 29.3.30.

Hydrocarbous gases, such as methane, ethane or other members of the paraffin series, unsaturated gaseous hydrocarbons such as ethylene or mixtures of both, containing sulphur or substances capable of supplying sulphur in the gaseous or vapour phase, are passed through a heated reaction zone at a high temperature and space velocity to convert all the sulphur into carbon disulphide without seriously affecting the yield of aromatic hydrocarbons furnished by the process. The source of the sulphur may be sulphur itself or hydrogen sulphide, which reacts under the conditions used. A suitable apparatus for carrying out the process is described in E.P. 349,958 (Abstr. 1085). S. F. B.

1164. Manufacture of Acetylene by Pyrogenation of Methane Gas. Société Industrielle Des Hydrocarbures et Derive's. E.P. 352,688, 16.7.31. Appl., 19.6.29.

Methane is partially converted into acetylene by passage through a tube heated to between 950° and 2000° C. at a pressure depending upon the temperature and varying from atmosphere at the lower limit of the temperature range to 25 mm. at the upper limit. The time of heating is determined by the temperature and pressure used, decreasing with rise of temperature and reduction of pressure, and the temperature by the relationship between the surface and volume of the pyrogenation apparatus. S. F. B.

1165. Absorption of Gas and/or Vapour in Oil. Standard Oil Dev. Co., assignees of H. J. Nichols, Jnr., and P. E. Kuhl. E.P. 353,079, 23.7.31. Appl., 2.5.29.

An absorption system is described of the kind suitable for absorbing gases and/or vapours in oil and means for separating the absorbed constituents from the oil, characterised by an improved absorption system, the operation of which is substantially automatic. The oil flowing into the top of the absorption vessel is controlled by the gas entering, this being carried out by the differential pressure on the two sides of an orifice plate controlling the compressed air which actuates the oil valve. S. F. B.

See also Abstracts Nos. 1223, 1239.

Motor Fuels.

1166. Airplane Fuel-Line Temperatures. O. C. Bridgeman, C. A. Ross and H. S. White. *J.S.A.E.*, 1931, **29**, 121-125.

The following equations are given for the calculation of the vapour-locking temperature of a gasoline with a conventional distillation curve.

(1) $t = 259 - 140 \log p_r$ where p_r is the Reid vapour pressure at 100° F. in lbs./sq. in.

t = vapour-locking temperature in ° F. at sea level.

(2) $t = 259 - 140 \log [(p_r \times 14{\cdot}7)p]$ where t = vapour-locking temperature at any altitude.

p = atmospheric pressure at the given altitude in lbs./sq. in.

(3) $$t = \frac{4.t_{10\%} \; 460 \log \left(\frac{p}{14{\cdot}7}\right)}{4 \log \left(\frac{p}{14{\cdot}7}\right)}$$

where t = vapour-locking temperature in °F.

$t_{10\%}$ = the 10% temperature in °F. determined by the A.S.T.M. Distillation method.

p = atmospheric pressure in lbs./sq. in. at the given altitude.

These give means for predicting the temperature and altitude under which a fuel of known Reid vapour pressure will give trouble from vapour lock. In order to calculate the permissible vapour pressure for freedom from vapour lock in an aeroplane, the probable fuel temperatures at various points in the fuel feed systems of five different aeroplanes used by the U.S. army and navy were determined during flight at different altitudes.

The results obtained are presented in the form of curves, the analysis of which leads to the following conclusions. Trouble might be anticipated under three sets of conditions. (1) In hot weather, the temperature of the fuel in the line leading to the carburettor may become sufficiently high to interrupt partially the fuel flow after the plane has landed and stood for a few minutes with the engine idling. Whilst the engine might run smoothly when throttled, engine stoppage probably would occur just after taking off, resulting in a crash. (2) If the fuel is close to the vapour-locking temperature before the plane leaves the ground, reduction in pressure and heating of the fuel in the line leading to the carburettor during a rapid climb to about 2000–5000 ft. altitude might lead to vapour-lock. This condition is probably limited to gravity-fuel systems. (3) As an airplane rises, the temperature of of the fuel does not decrease as rapidly as that of the air temperature. The atmospheric pressure continues to decrease and a point is reached where the vapour lock causes loss of power and irregular running. The latter are remedied by decreasing altitude so that these conditions limit the "ceiling" of the plane.

C. I. K.

1167. Benzol Refining. M. Kremer. *Petr. Zeit.*, 1931, **27**, 459-64.

The application of anhydrous aluminium chloride is described as a means of refining benzol in preference to sulphuric acid, which entails hydrocarbon losses. The aluminium chloride removes diolefines and converts unsaturated hydrocarbons into saturated hydrocarbons.

The research described was carried out on gas works benzol. 150 c.c. of the crude benzol were heated in a flask with 8–10 gms. of anhydrous aluminium chloride and then left to stand for a time. Hydrogen chloride was evolved and a black mass formed at the bottom of the flask. The flask was boiled on a water bath under a reflux for two hours, and on cooling the benzol was separated from the black mass, washed with dilute soda solution, then water-washed and distilled. The sulphuric acid test and bromine number of the resultant product showed a great improvement in the benzol. Even relatively small quantities such as 1–3% of aluminium chloride gave good results. Similarly good results were obtained with 10% by volume fractions of benzol.

A comparison of the results obtained with aluminium, iron and zinc chlorides shows that aluminium chloride is the most effective and zinc chloride the least effective. Aluminium chloride was also found to produce very good results in the refining of brown coal benzine. W. S. E. C.

1168. Synthetic Petrol. *Chem. Age*, 1931, **25**, 208.

In a paper before the Kaiser Wilhelm Institute at Mulheim-Ruhr, Dr. G. F. Fischer read a paper on the present production of synthetic petrol by the Fischer and Tropsch process.

An experimental plant has now been designed which produces 80% of the yields obtainable in the laboratory. Only the cheapest grades of gas can be considered as raw material, *e.g.*, mixture of coke-oven and blast furnace gas. The amount of heat required per lb. of petrol increases with the degree of liquefaction. Thus with a yield of 10% heating costs amount to 0·27d. per lb. of petrol and with a yield of 30% to more than 0·43d. per lb. To attain a good thermal efficiency the degree of liquefaction must be raised to 50%. Contact apparatus to produce this yield is very expensive, that required for a plant of capacity 10,000 cu. m. of gas per hour, costing £100,000. A plant of this size would produce 6000/7000 tons of high grade petrol per annum.

Further research should be carried out to find a cheaper contact material and a cheaper source of supply of gas. It is thought that the method used for solidifying fats can be applied to the synthesis of petrol. Gas mixtures that can be derived from methane are capable of producing much higher yields of petrol than the commercial gases previously tested. C. L. G.

1169. Fuels for Internal Combustion Engines. L. Rosenstein. E.P. 349,475, 18.5.31. Appl., 18.11.29.

The invention enumerates and describes the preparation of metallic compounds containing nitrogen and carbon which are anti-knock agents. Organo-metallo-cyanides are particularly useful, their anti-knock properties being due to both the metallic element and the cyanogen radical. Many of these compounds are soluble directly in hydrocarbons or in mixtures of these and alcohols. Their low volatility relative to that of the fuel is no disadvantage as the solid particles produce the same effect as the vapour. J. M. F.

1170. Purification of Crude Hydrocarbons and their Derivatives. I. G. Farbenind. A.-G. E.P. 352,164, 9.6.31. Appl., 17.5.30.

Crude hydrocarbons, such as benzol from coke ovens or gas plants, can be freed from certain undesirable constituents by treatment with maleic anhydride or acid followed by redistillation. The distillate is water-white, possesses little odour and gives only a slight yellow coloration with strong sulphuric acid. The residue left on distillation may be recrystallised to give methylene tetrahydrophthalic acid anhydride, m.-pt. 162° C. It is suggested that the crude residue may find uses in the preparation of resins by reaction with polyvalent alcohols, such as glycols or glycerine. S. F. B.

Lubricants and Wax.

1171. Evaluation of Viscosity Data. C. Walther. *Erdöl und Teer*, 1931, 7, 382–384.

The viscosity temperature curve of a lubricating oil is its most important property. To govern this, specifications usually require stated viscosities at three temperatures. This is unnecessary, however, since by the use of the viscosity temperature diagrams of McCoull, Herschel or Walther, the complete data can be obtained from measurements at two temperatures. If a certain other characteristic of the oil, such as the source, be known, a measurement at one temperature will suffice. The "viscosity index" of Dean and Davis and the "viscosity-gravity-constant" of Hill and Coats give criteria for determining the slope of the straight line obtained from the above-mentioned diagrams. Both these methods have certain disadvantages, so the author proposes the use of a "logarithmic temperature-factor," "m," in the formula,

$$\log \log (V_{kT}+0{\cdot}95) = -m (\log T - \log T_1) + \log \log (V_{kT1}+0{\cdot}95)$$

where T and T_1 are the absolute temperatures at which the viscosities V_{kT} and V_{kT1} (expressed as centistokes) are measured. "m" is a constant for the oil and may be written

$$m = a - b (\log \log V_{k50} + 0{\cdot}95);$$

values of a and b may be read off from the Walther viscosity temperature diagram, For, e.g., Russian oils,

$$m = 4{\cdot}24 - 13{\cdot}3 (\log \log V_{k50} + 0{\cdot}95).$$

By constructing a curve relating "m" and the viscosity-index, the latter can be read off as soon as "m" is known.

The complete behaviour of an oil with changing temperature is thus defined if the constant "m" and the viscosity at, say, 50° C. be stated. The constant holds good not only for a particular oil, but for all oils from the same source.

The remainder of the paper is devoted to a criticism of the views of Kadmer, Kyropoulos, Tauss and Sloane and Winning on this subject. E. B. E.

1172. Fluorescence in Mineral Oils. J. Tausz and C. Rabl. *Erdöl und Teer*, 1931, 7, 384.

The authors dispute the statement of Trautzl (cf. Abs. 511 and 807) that the removal of fluorescence by nitro-bodies is an optical and not a chemical phenomenon. Experiments with ultra-violet light cannot be used to prove the contention for visible light, since only ultra-violet light is completely absorbed by nitro-compounds. The fluorescence was still visible if a mineral oil was viewed through a thick layer of nitronaphthalene solution in toluol or aqueous picric acid. Even with a concentrated solution of the latter the green fluorescence could still be seen, while with less concentrated solutions the blue fluorescence was also visible, in spite of the light-absorbing properties of the solution. E. B. E.

1173. Solvent Extraction of Lubricating Oils. S. W. Ferris, E. R. Birkhimer and L. M. Henderson. *Ind. Eng. Chem.*, 1931, 23, 753–761.

The investigation was undertaken in order to ascertain the relative abilities of various solvents to separate lubricating oils into paraffinic and naphthenic factions. Previous work has been limited to several materials, particularly sulphur dioxide, but in the present investigation tests were carried out upon 110 substances as possible solvents and, of these, 21 have been subjected to exhaustive tests. Four different stocks were used for these tests, two being naphthenic (one a residium) and two semi-paraffinic in nature. The criterion

used for the difference in composition of the dissolved and undissolved fractions obtained with the various solvents was a value representing the difference in "paraffinicity" of the two fractions. The greater this difference the more selective the solvent. As a measure of "paraffinicity" the viscosity-gravity constant (a) was used; this was determined with the aid of the expression developed by Hill and Coats (*Ind. Eng. Chem.*, 1928, **20**, 641) as follows:—

$$a = \frac{10\,G - 1{\cdot}0752 \log (V - 38)}{10 - \log (v - 38)}$$

where V = Viscosity at 100° F. (Saybolt),
and G = Sp. gr. at 60° F.

or $$a = \frac{G - 0{\cdot}24 - 0{\cdot}22 \log (V^1 - 35{\cdot}5)}{0{\cdot}755}$$

where V^1 = Viscosity at 210° F. (Saybolt).

In this expression a high value for "a" indicates a high degree of "naphthenicity." The value ranges from 0·903 (for an extreme Gulf Coast type) to 0·807 (for an extreme Pennsylvania type). The results obtained indicated that the relationship

$$\frac{\text{volume of undissolved oil}}{\text{volume of solvent}} \quad vs. \quad \text{Viscosity gravity constant of the undissolved oil}$$

is practically independent of the temperature of extraction provided this is at least 25° C. below the critical solvent temperature of the solvent and stock. It can be assumed therefore that solvents can be satisfactorily compared on this basis.

Contrary to the indications from Hildebrand's work on solubility that the internal pressure and dielectric constant might be of value in selecting a solvent, it was found that no obvious relationship between these values and the suitability of a solvent existed. Indications were however obtained that the solvent action is profoundly affected by certain structural characteristics and the presence of certain groups, generally termed polar groups. Thus cyclic compounds were found to be better solvents than the open chain compounds, while an aliphatic side chain reduced the efficiency. The group attached to a ring obviously affects the solvent action, the order (in decreasing efficiency) being nitrite, nitro, aldehyde, acetate, hydroxyl and amine.

S. F. B.

1174. Deacidification of Lubricating Stocks. H. A. Young. *Oil and Gas J.*, 1931, **30** (15), 22.

The usual method of removal of acid sludge from treated lubricating stocks by settling generally results in the resolution of a portion of the sludge in the oil, and is disadvantageous from the point of view of the large storage capacity required, the great volume of oil required to be in process and the high oil losses in the sludge. Improvement is effected in many cases by the use of precipitating agents, such as water or alkali or finely divided solids, such as ground silica, fullers earth, Kieselguhr and various clays preferably applied in the agitators immediately after acid treatment. Centrifuging is also satisfactory in some cases, particularly with low gravity oils, but in general, owing to oil losses in the sludge with oils of high gravity, corrosion, fire hazard and lack of uniformity in results, it is not a practical solution.

Performance can be improved by paying attention to the agitation, viz., low treating temperature and use of mechanical agitation. Continuous withdrawal of the sludge pepper has been found essential to produce a sludge-free oil. The process described consists in the addition of filter aid to the oil from the

acid agitator and filtration through acid resisting asbestos cloth. Filtration is commenced by the usual precoating of the filter cloths. Filter aid is then added to the acid treated oil and the mixture pumped to the filters. At the end of the cycle the filter cake is blown with air or flue gas and the dry sludge cake removed. The sludge usually amounts to 28.5% of the oil. A typical cycle of operation is as follows: Filling filter, 5 mins.; precoating, 7 mins.; filtering, 260 mins.; draining, 3 mins.; blowing, 10 mins.; cleaning, 15 mins. The optimum conditions of settling and filtration are determined by laboratory control. Up to a certain point the rate of filtration can be increased by increasing the ratio of filter aid to compressible solids. The temperature of filtration should not be increased over the treating temperature to facilitate sludge removal.

Comparative tests of the colour of two different lubricating oils after settling and after filtration showed that whereas the optimum settling time for colour appeared to be 22 hrs., the true colour of the oil (after filtration) was best after only 8 hrs.

Details of the result of tests on Russian, Polish and Persian oils are given with an analysis of the cost of treating 1000 lb. per day of (*a*) light oil; (*b*) heavy oil; the total production charges, including overheads, being given as $0.089 per brl. for an oil of visc. 200 Say. Univ. at 130° F. and $0.157 per brl. for an oil of visc. 950 secs.

It is concluded that the economies effected by the filtering process should pay for the installation in from 6 months to a year.

Oils treated by this method show considerable improvements in quality (viz., complete absence of sludge, high stability and RE numbers, etc.), while economies are effected by the smaller tankage required and lower cost of chemicals, earth and handling charges. C. L. G.

1175. Motor Oil Consumption Gains Rapidly at High Engine Speed. *Oil and Gas J.*, 1931, **30** (1), 117.

9000 mile tests have been carried out on several makes of car, with the object of ascertaining the effect of speed on motor oil consumption. The tests were carried out at the Indianopolis Speedway under the supervision of the American Automobile Association acting for the Standard Oil Co. of Indiana. Cars used in the tests included Buick, Chevrolet, Ford, Cadillac and Willys-Knight, etc., and were all of 1931 type. The pre-diluted lubricating oils used were Iso-vis Nos. 30, 50 and 60, and the normal commercial lubricants used were Polarine No. 40 and a heavy Pennsylvanian motor oil of viscosity S.A.E. No. 50. In each case the procedure was: (1) Running in period of 2000 miles; (2) two runs of 1000 miles each, at a speed of 30 m.p.h. using a pre-diluted oil followed by a normal commercial lubricant; (3) five runs of 1000 miles each at a speed of 55 m.p.h. comprising one period on each of the pre-diluted oils and one period each on both the normal commercial lubricants. The result of the tests were: (1) Average consumption of oil of 1 quart per 1000 miles at a speed of 30 m.p.h.; (2) average consumption of oil of 6.9 quarts per 1000 miles at 55 m.p.h.; (3) reduction in oil consumption by about ⅓rd is obtained by using heavy summer oil in place of light winter oil; (4) gasoline mileage increased by 7% when using light winter oil as compared with heavy summer oil; (5) carbon deposited is smaller at the higher speed; (6) gasoline mileage decreased by running at higher speed, an average of about 13 m.p.g. being obtained at 30 m.p.h. and about 10.7 m.p.g. at 55 m.p.h.; (7) the viscosity of pre-diluted oils is not reduced in use; (8) the piston and cylinder wear was less than 0.001 in. for the entire 9000 miles.
R. A. E.

1176. The Field for Synthetic Lubricating Oils. F. W. Sullivan, V. Voorhees, P. T. Oak and D. P. Barnard. *J.S.A.E.*, 1931, **28**, 40–44.

In the course of an investigation into the composition of lubricating oils it has been found that oils of an extremely wide range of viscosity characteristics can be obtained by polymerisation of olefines with aluminium chloride. The viscosities, pour-points, etc., are given for the products of polymerisation of fifteen olefines from ethylene to cetene. The effect of the length of carbon chain in the starting material on the viscosity and viscosity index of the product is important, long saturated chains with a minimum of branching apparently yielding the best results: e.g., cetene with a straight chain of sixteen carbon atoms and a double bond in the 1–2 position gave the best results, considering both yield (85%) and viscosity index (138), while ethylene and pinene gave very poor results, the product from the latter having a viscosity index of −324.

During the last three years a large quantity of synthetic lubricating oil has been made from the olefines produced by cracking wax. These oils are more expensive than ordinary lubricating oils, owing to the complicated manufacturing process, but for some purposes, where cost is not a prime factor, their special properties make their use very advantageous. The viscosity index of these oils is better than for any natural oil, and yet, owing to the absence of wax, the pour-point is lower than for natural oils of similar viscosities. Tests have been carried out on the use of these oils in various units of car mechanism. In gear transmission it was found that at low temperatures the synthetic oil offered far less resistance to changing than similar natural oils. There was also a complete absence of channelling. Similarly in the steering mechanism, the response to human effort in cold weather was found to be far greater. Tests were also carried out with shock-absorber fluids, the high viscosity index desired being realised with these oils. The numerical results of these tests are tabulated and charted.

As regards the use in internal-combustion engines, the low Conradson coke values and absence of asphalt-forming constituents are advantageous, while the good viscosity temperature characteristics are especially valuable for certain types of aircraft and motor-coach engines.

Finally the use of these oils in certain cases allows the designer greater freedom. Although they have been produced in commercial quantities, the oils must still be regarded as in the development stage. E. B. E.

1177. Lubricating Oils from Crude Oils by Treatment with Hydrogen at High Temperatures and Pressures. Standard Oil Dev. Co. E.P. 352,775. 16.7.31. Appl., 14.8.29.

Lubricating oils are produced from relatively heavy hydrocarbon oil by passing the latter and gas rich in free hydrogen in opposite directions through a deep bed of a catalytic material. The temperature at which the operation is carried out should be within the approximate range 700–825° F., while the pressure is maintained from 50 atmos. to above 100 atmos. The temperature control is important, being regulated so that the formation of light oil is relatively slow. Various examples of catalysts are given. W. D. S.

1178. Lubricant. G. D. Peters & Co. E.P. 353,295. 23.7.31. Appl., 26.8.30.

A lubricant is described which is particularly adapted for lubrication of, and for removing rust from, leaf springs, and also for any close fitting bearing surfaces having frictional contact. This penetrating lubricant consists of

light petroleum oil (100° Saybolt at 100° F.), suspended graphite and a small proportion of thinner including a non-inflammable volatile chlorinated hydrocarbon. W. D. S.

1179. Cleaning of Lubricating Oil in Internal Combustion Engines. Ariel Works, Ltd., and J. Y. Sangster. E.P. 353,565. 30.7.31. Appl., 10.5.30.

A lubricating-oil cleaner of the centrifugal type is described for use in connection with internal-combustion and other engines. The fly-wheel contains chambering in radial relation to its axis of rotation, and is connected with the lubricating circuit by inlet and outlet orifices so that when the wheel is revolving, any impurities suspended in the oil are thrown centrifugally towards the outer end, whilst the cleansed oil is allowed to pass to the parts of the engine requiring lubrication. In this system the oil supply passage of the intercepting chamber in the fly-wheel is connected to the bore of a bored stem or conduit pipe located in the chamber. The outlet of this stem into the chamber is so disposed between the chamber ends that, on the fly-wheel coming to rest, any intercepted impurities gravitating into the oil-intake end of the chamber are trapped or prevented from returning to the lubricating circuit. W. D. S.

1180. Dewaxing Oil. S. W. Ferris. U.S.P. 1,816,367. 28.7.31. Appl., 9.3.28.

The wax-containing oil is first treated with a sludge-forming acid at normal temperatures and the oil removed from the sludge. The oil is then chilled to a temperature at which at least a portion of the wax content will thereby form solid particles (+20° F.). While cold the oil is treated with a sludge-forming acid and the mixture allowed to settle. The wax and resulting acid sludge are removed from the oil at the temperature of treatment and the separated oil neutralised. A diluent may be employed. W. D. S.

1181. Refining Lubricating Oils. W. D. Rial. U.S.P. 1,816,872. 4.8.31. Appl., 13.2.29.

Lubricating oil stocks derived from an asphalt or mixed base petroleum oil are refined so as to produce an oil which will not emulsify with water or a water solution of caustic soda. The stocks are separated by vacuum distillation, and immediately, with the exclusion of air, condensed and redistilled. The redistilled stock is again condensed with the exclusion of air and immediately treated in the liquid phase with a water solution of KOH in the presence of steam in order to remove the oxidisable constituents. W. D. S.

1182. Reconditioning Oils. J. G. Ford and Westinghouse Electric Co. U.S.P. 1,818,218. 11.8.31. Appl., 6.8.27.

A process is described for refining mineral oils that have been used in transformers or other oil filled apparatus. The oil is treated with 2 to 4% of fuming H_2SO_4 mixed with fuller's earth and the mixture agitated. The water, oxidised hydrocarbon products and residual H_2SO_4 are allowed to separate and removed. W. D. S.

1183. Hot Filtration of Decolourised Oils. P. W. Prutzman. U.S.P. 1,818,274. 11.8.31. Appl., 20.4.27.

A continuous method of decolourising mineral lubricating oils with a solid absorbent decolourant is described. The oil and decolourant are heated to a temperature, not less than 350° F., but below substantial cracking temperatures, at which vapours are evolved. The oil vapour and any accompanying

steam are separated from the mixture while substantially preventing any reduction of temperature and the devaporised mixture cooled and filtered to remove spent decolourant. The separated spent adsorbent, prior to exposure to the atmosphere, is cooled by the passage of a non-oxidising fluid at a temperature lower than that of the adsorbent—e.g., steam. W. D. S.

1184. Refining Hydrocarbon Oil. A. L. Lyman and Standard Oil Co. (Calif.). U.S.P. 1,818,488, 11.8.31. Appl., 6.10.26.

Hydrocarbon lubricating oils are refined by agitation with 1–1½ lb. of 66° Bé. acid per gal. in a single treatment, the sludge formed being removed by passage through a bed of clay at below 140° F., and the oil finished by a separate decolorising operation wherein the oil is treated with a decolorising clay. The sludge formed is of a granular form, and the major portion may be settled out before passage through the clay bed. W. D. S.

See also Abstract No. 1238.

Fuel Oil.

1185. Fuel Oil Specifications. *Oil and Gas J.*, 1931, **30** (14), 97.

In August, 1931, Chicago Burning Oil Heat Institute members adopted a code of ethics governing the sale and distribution of their products. They also recognised the following designations and specifications for fuel oil to be distributed within Cook County, Illinois :—

Grade.	Fl. pt. °F.	Colour.	Saybolt Viscosity at 100° F. secs. max.
Distillate No. 1	165	lt. straw	—
Lt. straw gas oil, No. 2 ..	190	straw	—
Dark gas oil, No. 3	190	dark	—
Fuel Oil, No. 4	200	,,	55
Fuel Oil, No. 5	225	,,	125
Fuel oil, 18–22, No. 6 ..	290	,,	500

Cold tests of 0° F. are specified in all cases, and for the first two oils respectively maximum 10% distillation temperatures of 420° F. and 440° F., and E.P. of 600° F. and 650° F. are demanded. A sweet odour is required in the case of the first two oils and a good odour in the case of the other four oils. R. A. E.

1186. Oil Fuel Burners. Babcock & Wilcox, Ltd. and A. Frankish. E.P. 352,816, 16.7.31. Appl., 20.9.30.

The invention relates to a safety shut-off device for oil fuel burners which automatically shuts off the fuel when the burner is being changed.

A small valve is held off its seat by the head of a spindle which holds the cover over the burner head. As this spindle is rotated to allow the removal of the cover, the spring behind the valve closes it, and thus shuts off the hot oil supply. Also a socket, fitting over the valve cage, is so arranged that after the valve is closed the socket has still to be drawn along the greater portion of the sleeve round the valve.

Thus no hot oil can escape into the boiler rooms to injure the operator. J. M. F.

Fluxes and Asphalts.

1187. Standard Sieves. P. E. Spielmann. *Roads and Roads Const.*, 1931, **9**, 233–234.

This article gives in concise form a discussion of the various series of sieves which have existed, the old and new I.M.M. series, the A.S.T.M., and the "Test Sieves" covered by the new B.E.S.A. specification No. 410, 1931. The historical side of the development of each set, together with a detailed comparison of their merits and a table giving the sizes of the apertures in each set of sieves, should be of value to those handling materials for road construction. C. I. K.

1188. New Materials and Processes in Bituminous Road-Making. R. Wilhelmi. *Erdöl und Teer.* 1931, **7**, 319–321 and 337–339.

This is from a brochure published by the Association of Austrian Road Companies of Vienna, and consists of an extensive summary of recent work on Asphalt and Tar road-making.

Much attention is paid to emulsions and methods of manufacture, many references being given to the German patent literature. E. B. E.

See also Abstracts Nos. 1236, 1237.

Special Products.

1189. The Petroleum Industry as a Supply for Industrial Solvents. J. A. Park, J.S.C.I., 1931, **50**, 620–622.

Light petroleum naphthas are used in the dry-cleaning industry, the manufacture of varnishes and paints, and as solvents in crystallising out wax from Lubricating Oils. The chemical solvents produced from petroleum hydrocarbons are the following: *Alcohol Group*—Synthetic amyl alcohol containing five of the eight amyl alcohols, prepared from n-pentane and isopentane by treatment with chlorine; ethanol and the secondary alcohols from isopropanol up to secondary hexanol are formed by absorption of olefines with sulphuric acid. *Ether Group*—Ethyl and isopropyl ethers are formed in conjunction with the preparation of alcohols; ethers of ethylene glycol are prepared by treating an alcohol with ethylene oxide. Esters are produced by the action of a fatty acid and an alcohol, with vapour phase esterification, or, by treatment of alkyl sulphuric ester with the calcium salt of a fatty acid. Chloro-compounds, such as ethylene dichloride, are widely used as extraction solvents.

The majority of industrial solvents now produced from petroleum hydrocarbons are utilised by the nitro-cellulose industry, the esters being the most satisfactory solvents for nitro-cellulose available at present. Amongst other new developments pertaining to the use of industrial solvents from petroleum is the extraction of acetic acid from pyroligneous acid by means of ethyl or isopropyl ether, 98–99% recovery being claimed. Isopropyl ether, a new industrial solvent, compared with ethyl ether, has a lower vapour pressure, higher boiling point, higher flash point, is not so soluble in water, and possesses somewhat higher solvent powers.

An interesting survey is made regarding the use of different solvents in the lacquer industry, and the cause of such effects as "blushing," "pinholing," etc. W. A. W.

1190. Catalytic Condensation of Amylenes. A. Mailhe and Renaudie. *Compt. rend.*, 1931, **192**, 561–3.

Trimethylethylene and pentene-2 were passed over silica gel contained in a quartz tube heated to 670° C. The former gave a gas containing 47% olefine hydrocarbons (ethylene and propylene), 38% methane and 7% hydrogen; an oil and tar remained which on fractionation gave higher olefines, aromatic hydrocarbons and a residue melting at 80° C. The gas from the pentene-2 contained 52% propylene and butylene, 34% methane and 6% hydrogen; the oil and tar yield higher olefines, aromatic hydrocarbons and a residue melting at 53° C. Various physical properties of the fractions are given.

S. F. B.

1191. Oil for Printers' Ink. *Oil and Gas J.*, 1931, **30** (12), 55.

Modern printing ink is a development of the formula of Cannefarius, published in 1660, which consisted of 1 lb. of varnish made from linseed oil and Juniper gum incorporated with 1 oz. of smoke black and brought to the proper consistency over a slow fire.

Nowadays varnish made up with beeswax, soap, shellac, turpentine, etc., is used, the carbon being the carbon black prepared from natural gas. Cheaper grades such as are used by newspapers are made from 10% carbon black and 90% mineral oil. The oil is usually a blend of a semi-asphaltic cylinder stock with a small quantity of light neutral oil. A typical oil had a gravity A.P.I. 26.1°, flashpoint open cup 385° F., viscosity Saybolt Universal 55 secs. at 210° F., sulphur less than 0.5% and pour point 20° F. An oil of this kind should leave no trace on the under side of a piece of printed paper and should have no tendency to creep and produce a brownish tinge along the edges of letters or designs. Rapid absorption by the paper is necessary, for a desirable news ink dries almost entirely by absorption and to a very small extent by evaporation.

C. L. G.

1192. Separation of the Oxidation Products of Solid Hydrocarbons, Waxes and like Materials. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 352,512, 13.7.31. Appl., 13.1.30.

The separation of oxygenated products of an incomplete oxidation of solid hydrocarbons, waxes and like materials from unattacked initial materials, or of oxygenated products from each other, is accomplished by centrifugal pressing. A diluent may be used if desired. A temperature is employed such that the constituents are removed in a fluid state.

W. D. S.

1193. Extraction of Ethylene Glycol. Chemische Fabrik Kulk G.m.b.H. and H. Ochme. E.P. 352,651, 16.7.31. Appl., 24.5.30.

Glycol is extracted from dilute solutions containing calcium or magnesium chlorides by evaporation in a vacuum at temperatures between 250–400° C. with simultaneous use of agitators or other mechanical means. The water is driven off almost entirely and the aqueous glycol-calcium or magnesium chloride solution left further dried in a thin layer in a vacuum drier at about 170° C. The glycol is finally driven off at 320° C. in the drier, the anhydrous chloride being obtained as a by-product.

S. F. B.

1194. Manufacture and Production of Alcohols. I. G. Farbenind. A.-G. E.P. 353,047, 10.7.31. Appl., 10.4.30.

A process for the manufacture and production of oxidation products consisting mainly of alcohols which consists in bringing hydrocarbons, solid or liquid, having a boiling point over 180° C., in a liquid state into contact

with air or other gaseous oxidising agents at atmospheric pressure, the oxidation reaction being carried out in the presence of acids having a dissociation constant between 2.5×10^{-4} and 2×10^{-13}, for example organic acids, such as formic, acetic, propionic, maleic, benzoic acids and weak inorganic acids such as boric acid or its anhydride, antimony pentoxide, stannic acid, etc. The quantity of acid added must be at least 2% by weight of the hydrocarbon to be oxidised, and the operation is stopped when the saponification value reaches 70. S. F. B.

1195. Decomposition of Gaseous Hydrocarbons to Obtain Hydrogen and a High Grade Carbon Black. Société d'Etudes et Realisation dite "Ereal." E.P., 353,100, 23.7.31. Appl., 20.4.29.

Gaseous hydrocarbon mixtures are decomposed to give hydrogen and high grade carbon black by passage through an incandescent fuel mass, e.g., coke contained in a chamber under such operating conditions that the temperature of emergence is between 600° and 700° C. The carbon black carried out of the reaction chamber by the resulting gases is settled out in a special chamber or removed in any other way desired. The coke is raised to the required temperature, 1000–1200° C., by air blowing; when it has reached this temperature the air is replaced by the gas to be cracked and this operation allowed to proceed until the temperature reaches 700–800° C. when the air blowing is repeated. S. F. B.

1196. Carbonaceous Product. R. T. Goodwin, assr. to Standard Oil Dev. Co. U.S.P., 1,812,589, 30.6.31. Appl., 18.2.26.

The patent describes a "new article of manufacture" which is precipitated by a dilute acidic reagent from heavy residues from cracking. The product contains about 25% of asphaltic matter and 75% of non-asphaltic material and is characterised by a fusing point between 160° and 230° C. and a solubility in carbon disulphide of 15% to 30%. W. H. T.

Refining and Refinery Plant.

1197. Petroleum Refining: Progress of the Canadian Industry. *Chem. Trade J.*, 1931, **89**, 210.

The report of the Dominion Bureau of Statistics at Ottawa on the Petroleum Products Industry gives the value of the products obtained in 1930 as $91,787,205, a decrease of 8% on the 1929 record production. There are fifteen refineries in Canada with a total throughput of 1,064,434,974 gal. of crude oil, of which 65% is of United States origin, 30% of South American and 5% of Canadian origin. Only one refinery in Alberta operates entirely on Canadian crude.

Imperial Oil, Ltd., has just completed at Sarnia, Ontario, a large new unit for the production of high-grade motor oil by a process developed by Dr. Stratford, which it is claimed embodies the almost complete removal of carbon and gum forming constituents. A debutanising plant for gasoline is also to be constructed.

The Northwest Stellarene Co. at Coutts, Alberta, have recently installed a vapour phase cracking installation, based on an adaption of the Leamon process. The crude worked up is obtained from the Red Coulee field, which is partly in Montana and partly in Alberta.

Statistics of the capitalisation of the petroleum industry and value of products obtained are given with the locations of the various refineries and details of the amounts of the different products obtained from the crudes worked up. C. L. G.

1198. Refining Mineral Oils. W. W. Triggs., assr. to Richfield Oil Co. E.P. 353,054, 15.7.31. Appl., 15.4.30. Refining Hydrocarbon Oils. Richfield Oil Co. E.P., 353,055, 15.7.31. Appl., 15.4.30.

A process of refining mineral oils is described which comprises vaporising the oil, contacting the vapours with a hot aqueous solution (at 300–400° F.) of a salt or salts of one or more of the metals, zinc, cadmium and mercury, and then condensing the vapour. The solution may be one of a zinc salt (50–85%) containing zinc hydroxide to the extent of 0·5 to 3% by weight, and is maintained in a fluid condition by the addition of water. W. H. T.

1199. Fractional Extraction of Mineral Oils. H. G. C. Fairweather, assr. to the Atlantic Refining Co. E.P. 353,3072, 3.7.31. Appl., 5.9.30.

Mineral oils containing both paraffinic and naphthenic hydrocarbons can be extracted with nitrobenzene or other nitro compounds of an aromatic hydrocarbon—e.g., nitrotoluene, mononitro-naphthalene, etc., to give an extract containing more naphthene hydrocarbons than the original oil and a residue containing more paraffins than the original oil. The process is not limited to one extraction, and the intermediate portions possess properties in between the two extremes—i.e., the undissolved portion and the first extract. The process is carried out by mixing the oil to be treated with the solvent at 60° C., and then cooling with gentle agitation to about 10° C., when the agitation is discontinued and the whole allowed to settle. The two layers are separated and, if desired, the process repeated with the upper, undissolved layer. The solvent is removed from both upper and lower layers by distillation with steam under reduced pressure. If necessary the upper layer may be chilled to about −20° C., diluted with naphtha and the separated wax removed in the usual manner. The naphtha is removed from the oil layer by distillation. S. F. B.

1200. Refining Hydrocarbons. F. A. Apgar, assr. to Sinclair Refining Co. U.S.P. 1,812,426, 30.6.31. Appl., 29.2.28.

An improvement in the vapour phase refining of hydrocarbons by passing them through a charge of an adsorptive catalyst consists in the passage of vapours through the catalyst in admixture with a relatively inactive gaseous diluent, and the control of the vapour-gas ratio to agree with increases or decreases in the activity of the adsorbent. W. H. T.

1201. Apparatus for Refining Oils. C. W. Stratford. U.S.P. 1,812,612, 30.6.31. Appl., 24.11.26.

A still is described which is adapted to maintain a constant level of oil therein and fitted with a circulating device. The latter is capable of drawing off oil and discharging it under pressure in a plurality of separate streams against the inner heated surface of the still. By this means, heat exchange is increased and a rapid circulation of the oil is induced. A draw-off is connected to the circulation means for removing treated oil from the body of the still. W. H. T.

1202. Decolorising Hydrocarbon Oils. H. Lowery and R. S. Lane and Standard Oil Co. (Ind.). U.S.P. 1,813,628, 7.7.31. Appl., 27.1.27.

Hydrocarbon oils are decolorised by the following process. The oil is dissolved in a lower boiling distillate, the solution treated with sulphuric acid and the sour oil mixed with decolorising clay. The oil-clay mixture

is passed through a pipe-still, where it is heated to between 650° and 700° F. From thence the heated mixture passes to an enlarged chamber to which steam is supplied, whereby the lighter fractions are removed. The unvaporised oil and admixed clay is filtered between about 250° and 400° F. The oilclay mixture in the pipe-still, though heated to cracking temperature, is moved at such a velocity that cracking is avoided. W. D. S.

1203. Purifying Petroleum Products. H. G. Smith and Gulf Refining Co. U.S.P. 1,813,642, 7.7.31. Appl., 16.6.26.

The final purification of petroleum products to remove taste and odour is accomplished by repeatedly warming the product with a plurality of portions of active aluminium chloride with intermediate removal of sludge. The treated material is washed with a hot dilute solution of H_2SO_4, dried, filtered with clay, and submitted to reduction with fire and steam. The clay treatment is then repeated. W. D. S.

1204. System for Treating Petroleum Oils. C. W. Stratford. U.S.P. 1,815,366, 21.7.31. Appl., 26.11.26. Divided, 12.6.29.

A closed method for treating petroleum oils comprising the treatment of the oil with a chemical in a number of rapid contact stages alternating with stages of centrifugal separation, the chemical flowing counter-current to the oil. A flow sheet and drawings of the contactors and separators are given. S. F. B.

1205. Sweetening Petroleum Distillates. L. M. Henderson and H. C. Cowles, Junr. U.S.P. 1,815,563, 21.7.31. Appl., 15.3.28.

Sour petroleum distillates, either primary or cracked, can be sweetened by treatment either in batch or continuous process with a suitable reagent, particularly a copper salt, in aqueous solution. The reagent is revivified after separation by treatment with oxygen or an oxygen-containing gas. A feature of the invention is that the aqueous reagent is maintained in a neutral or substantially neutral condition by the addition of a buffer salt or mixture of such salts. The absence of sludge in the spent reagent considerably facilitates the handling thereof and dispenses with the problem of sludge disposal. A suitable reagent is stated to be an aqueous solution containing 20% cupric chloride and 0·75% borax or sodium tetraborate, and the proportions suggested are 500 parts by volume of gasoline to 200 parts of the reagent. After vigorous agitation for a few minutes, during which time the distillate becomes sweetened, the reagent is drawn off and revivified by air blowing. It is then ready for further use. S. F. B.

1206. Oil Purifying System. D. E. Pierce, assr. to Atlantic Refining Co. U.S.P. 1,816,294, 28.7.31. Appl., 5.8.26.

A process is described for the removal of gum forming and colour-imparting bodies from hydrocarbon oils. It comprises passing the oil in vapour phase upwardly counter-current to and in contact with a descending stream of liquid hydrocarbon oil. The latter is free from gum and colour importing bodies, but is capable of removing these bodies from the vapours without causing polymerisation or undergoing any change itself. The spent oil is continuously removed from contact with the vapours, treated with sulphuric acid to effect removal of the gum forming and colour imparting bodies, and continuously returned into contact with the vapours. W. H. T.

1207. Process of Steam Distillation. J. G. Glasgow. U.S.P. 1,817,250, 4.8.31. Appl., 11.5.26.

The process comprises pre-heating of the raw feed by admixture of hot residuum, stepping up the temperature of the mixture to supply enough heat for the vaporisation of the desired fraction, introducing steam into the still under conditions whereby it reduces the boiling point of the material undergoing distillation, subjecting the vapours to partial condensation and returning the condensate to the still.

The gasoline vapours are condensed whilst the still feed is controlled to a temperature below that which will injuriously affect the products of distillation, but sufficient to supply the heat necessary for distillation of the desired fractions. R. P.

1208. Purification of Benzenoid Hydrocarbon Oils. P. Damm, Assr. to the Koppers Co. U.S.P. 1,817,804, 4.8.31. Appl., 30.8.30.

Benzenoid hydrocarbons are purified by treatment with sulphuric acid of such a strength that the products derived from the undesirable constituents are not rendered insoluble in the hydrocarbon layer after neutralisation. It is claimed that such a treatment allows valuable products to be recovered which would otherwise be converted into tar-like waste material. The strength of the acid used may vary from 60° to 65·5° Bé., depending upon the material to be treated, while the temperature during treatment should not exceed 50° C. Three layers are formed. The lower consists of acid which is sufficiently clean to be used for other purposes without any recovery treatment. The intermediate layer, after the acid layer has been removed, will re-dissolve on neutralisation; it contains valuable resins which may be recovered by distillation. S. F. B.

1209. Filtration of Tars. F. J. Gainer, Assr. to Standard Oil Co. (Ind.). U.S.P. 1,819,745, 18.8.31. Appl., 5.7.28.

Petroleum residues such as pressure tars from a cracking plant are filtered at high temperatures and under pressure through an asbestos filter medium which has been coated with graphite. Very finely divided dry graphite is rubbed into the outer surface of the asbestos leaves with the bare hand; the graphite prolongs the life of the asbestos plates. The liquid portion of the tar is forced through the filter into perforated tubes from which it flows through a trough into pipes. When the rate of filtration becomes slow owing to a collection of solid material in the chamber the latter is opened and the solid cake is removed. The filter leaves are found to need cleaning and further graphite applied after 20–25 days' use. W. S. E. C.

Cracking.

1210. New Process of Distilling Petroleum Oils. R. Fussteig. *Petr. Zeit.*, 1931, **27**, 520–22.

The "Weymann System" of continuous cracking distillation is described. The plant comprises a cracking still consisting of two tube banks with an atomiser between them. In the first bank the oil is subjected to a preliminary cracking operation in the liquid phase at the desired temperature and under pressure. The hydrocarbons are vaporised in the atomiser cylinder under reduced pressure and cracking is completed in the second tube bank in the vapour phase. The plant is recommended on its advantages of small initial outlay, economic operation on a small scale, lack of carbon deposits and high yields. W. S. E. C.

1211. Conversion of Liquid Carbonaceous Materials into Low Boiling Point Products. H. C. Wade. E.P. 352,448, 1.7.31. Appl., 1.3.30. Conv. (U.S.A.), 22.5.29.

This is a process which claims to produce synthetic liquid hydrocarbons and comprises vaporising a heavy hydrocarbon stock under heat and leading the vapours in a spiral path, during the heat treatment, at a high velocity without pressure through a catalyst; passing water vapour through the catalyst simultaneously thereby evolving free hydrogen which combines with the free carbon. R. P.

1212. Treating Oils. J. C. Black. E.P. 353,148, 23.7.31. Appl., 20.5.30.

A continuous process for converting heavy oils into lighter ones, in which the former are first heated in order to distil off a portion. Some of the condensate formed is cracked in a heated coil while a further quantity of the condensate at a temperature substantially lower than the cracking temperature is introduced into the cracking coil in the region of the hottest point in which the rate of cracking is unduly rapid. W. H. T.

1213. Cracking Oil. R. C. Osterstrom and C. R. Wagner. E.P. 353,230., 23.7.31. Appl., 8.7.30.

A method is described of cracking oil in the vapour phase by first removing from the oil those fractions which do not vaporise without molecular decomposition. Before cracking, the vapours are dried by passage through a superheating zone. This zone comprises tubes of relatively great cross section as compared with those of the cracking and vaporising zones. W. H. T.

1214. Cracking Hydrocarbon Oil. A. E. White, Assr. to Texas Co. E.P. 353,276, 23.7.31. Appl., 12.8.30.

The process comprises the distillation of oil, which has been subjected to a cracking reaction, to form a heavy residual oil and a vapour fraction. The latter is fractionated to give a light fraction, and intermediate fraction and a heavier fraction. The intermediate fraction is blended with the heavy residual oil to form a fuel oil, while the heavier fraction is conducted to a cracking zone. W. H. T.

1215. Manufacturing High Compression Automotive Distillate. Automotive Distillate Corpn. U.S.P. 1,810,574, 16.6.31. Appl., 14.6.28.

The invention provides improved means for carrying on several cracking processes on a given oil in such a manner that coking in the furnace tubes is eliminated, and a better product produced, without the use of an expansion chamber. The arrangement of the plant allows accurate control of the process whilst utilising the minimum number of elements and the minimum amount of heat. J. M. F.

1216. Apparatus for Cracking Hydrocarbons. N. E. Loomis. U.S.P. 1,812,658., 30.6.31. Appl., 22.7.25.

An apparatus is claimed consisting of a cracking coil; a soaking drum; a still; a bubble tower; a vapour heat exchanger in the tower and means for passing feed oil through this exchanger; a tar heat exchanger connected to the still with means for passing the preheated feed through the tar heat exchanger and thence to the still.

The heavy condensate formed in the tower is collected and passed through the coil and soaking drum to the still. R. P.

1217. Apparatus for Decomposing Hydrocarbon Oils. H. T. Darlington. U.S.P. 1,813,117, 7.7.31. Appl., 24.6.29.

One of the objects of the invention is to provide an improved heating unit for cracking oils. Further objects are specified.

A new and improved apparatus is claimed comprising a vertically disposed chamber and a heating unit composed of a header in the form of a vertically disposed cylindrical chamber standing in a substantially parallel and spaced relation to the main chamber, a group of heating and circulating tubes extending from one end of the said heater and communicating with a corresponding portion of the main chamber, and a second group of heating and circulating tubes extending from the other end of the said heater and communicating with a corresponding portion of the main chamber and means for heating the said tubes.

In addition to the above there is provided mechanical means for introducing, controlling, and directing the circulation of oils through the said unit; means are also provided for the heating of the tubes; moreover, the main chamber extends below the tubes for the purpose of providing a pool for accumulation of heavy materials. R. P.

1218. Transformation of Petroleum Hydrocarbons into Gasoline. U. S. Jenkins, Assr. to Jenkins Petroleum Process Co. U.S.P. 1,814,042., 14.7.31. Appl., 16.11.28.

A cracking process is described in which an adsorptive binder, such as hydrated lime, is introduced to the body of oil undergoing cracking. The carbon reaction products are subsequently withdrawn associated with this binder, and a coal-like substance is obtained. H. C. R.

1219. Cracking Still. W. T. Hancock. U.S.P., 1,814,249., 14.7.31. Appl., 21.8.29.

A cracking still, consisting of a plurality of tubes, is described in which each tube is fitted with a central shaft on which are mounted at an angle a series of ball-confining discs. Metallic balls between these discs serve, on rotation of the shaft, to hammer and scrub the walls of the cracking tube with the result that solid matter is prevented from depositing thereon. H. C. R.

1220. Decomposing Organic Materials. R. J. Dearborn and G. W. Gray, Assrs. to The Texas Co. U.S.P., 1,814,621, 14.7.31. Appl., 20.4.26.

A method is described for the decomposition of organic substances by the application of heat and pressure, preferably in the liquid phase, with the aid of an oxidising gas. Water soluble oxygen-containing substances are obtained in addition to the water insoluble fractions suitable for motor fuel. H. C. R.

1221. Digester for Cracking Hydrocarbon Oils. J. C. Black, Assr. to Gasoline Products Co. U.S.P., 1,815,428, 21.7.31. Appl., 25.10.22.

A digester or reaction chamber for use in cracking hydrocarbons is described consisting of a tubular metallic chamber lined with a corrosion resisting lining such as a silicon carbon alloy or a silicon iron alloy. Sketches and a detailed description of the apparatus are appended. It is claimed that the apparatus will withstand temperatures up to 1000° F. at a pressure of 1000 lbs./sq. in. H. C. R.

1222. Apparatus for Treating Hydrocarbons. R. Cross, Assr. to the Cross Development Corpn. U.S.P. 1,816,827, 4.8.31. Appl., 20.1.26. Divided 5.4.26.

The apparatus described consists essentially of a Cross cracking plant with means for leading off the vapours through the towers containing refining agents and then into a rectifying column. The towers are duplicated so that operation may be made continuous by the use of either pair of towers or by replacing any one when the refining agent becomes spent. These towers are well lagged to prevent heat losses. The first, through which the vapour passes, contains metallic compounds such as copper salts, copper oxide, metallic copper, iron or salts of metals having an affinity for sulphur compounds, while the second contains an adsorbent material such as Fuller's earth, gelatinised clay, charcoal, etc. It is claimed that this apparatus gives sweet gasoline without further treatment. S. F. B.

1223. Process for Producing Light Hydrocarbons. H. Butler. U.S.P. 1,817,672, 4.8.31. Appl., 27.12.26.

Heretofore it has not been found possible to convert into petrol all the petroleum products introduced into the cracking still. It has been proposed to produce gasoline from natural gas by absorption methods. In these absorption processes only a small proportion of the gas is converted into liquid products.

This invention provides a process whereby high boiling hydrocarbons are cracked in liquid or vapour phase and the light hydrocarbons from natural gas are passed into the cracking zone during the cracking operation, and consists of an absorber for natural gas; means for distilling absorbed hydrocarbons and for compressing and cooling the products; means for introducing the uncondensed gases into a cracking plant; means for cooling the resulting vapours, and means for recirculating the uncondensed vapours. R. P.

1224. Oil Vaporiser and Converter. C. B. Watson and A. E. Harnsberger. U.S.P. 1,818,512, 11.8.31. Appl., 21.2.28.

This invention provides an oil furnace which is particularly applicable to vapour phase cracking. It is necessary to provide a highly efficient type of furnace to secure the operating temperature and at the same time make the most efficient use of the heat generated, in order to operate the system economically as far as fuel consumption is concerned.

In the present invention the claims are for a furnace composed of primary and secondary chambers constructed so that the furnace gases have an upward movement through the primary and a downward movement through the secondary chamber. A single row of heating tubes are arranged in the primary chamber and a bank of tubes in the secondary chamber. The flow of oil is parallel with the flow of furnace gases in the secondary chamber, but counter-current to the flow of furnace gases in the primary chamber. R. P.

1225. Cracking of Oil Vapours into Vapour Phase. J. B. Weaver and C. R. Wagner, Assrs. to Gyro Process Co. U.S.P. 1,818,513, 11.8.31. Appl., 17.2.28.

The invention refers particularly to high temperature processes wherein the oil is maintained in the vapour phase at low pressures, and a method is claimed whereby gasoline produced by this method may be refined for the removal of those substances which are undesirable. These substances produce gum and

discoloration on standing. To refine this type of gasoline it is proposed to introduce the vapours of such cracked oils admixed with steam into intimate contact with a finely-divided, adsorbent catalyst, the steam being present in such proportion as to cause polymerisation of about one-fourth of the total unsaturates which would be polymerised without the presence of steam, the remaining unsaturates being unpolymerised and thus present in desirable increased amount over that originally present in the raw material charged to the polymerising zone. R. P.

1226. Process for Converting Hydrocarbons. W. M. Cross. U.S.P. 1,818,674, 11.8.31. Appl., 11.3.25.

The objects of the invention are to provide a process in which oil is heated to a cracking temperature in a coil and transferred to a zone where polymerisation and cracking is permitted to continue to the desired extent. This heating and conversion takes place under high pressure, that is, sufficient to prevent substantial vaporisation during the heating or cracking period. The cracked products are cooled, and while in this condition are separated into liquid and gas. The liquid or synthetic crude is refined and utilised as a cooling medium for the highly-heated oil discharged from the reaction stage, whereby a liquid end-point distillate may be obtained by dephlegmation.

Seven claims are made for the invention, including a process for converting hydrocarbons comprising the steps of treating and digesting the oil at relatively high temperatures and pressures, withdrawing the cracked products in highly-heated condition, cooling the cracked products in a heat exchanger to distil off the lighter constituents, and passing the reheated products to stages of vaporisation, dephlegmation, and final condensation, with recycling of the bottoms to the conversion zone. R. P.

1227. Treating Heavy Oils for the Production of Light Oils by the Employment of Metallic Halides. W. H. Sheffler, Assr. to the Standard Oil Co. (Calif.). U.S.P. 1,819,006, 18.8.31. Appl., 11.8.26.

The conversion of higher b.-pt. oils into products of lower b. pts., such as gasoline, kerosine, etc., by distillation in the presence of aluminium chloride or other metallic halides may advantageously be carried out under increased pressure, whereby the time of reaction is considerably reduced. The pressure employed should be sufficient to maintain the main part of the unconverted oils in the liquid phase, but insufficient to prevent vaporisation of the lighter oils produced. For example, it is suggested that the pressure employed be sufficient to maintain the temperature of the oils at approximately 50° to 70° F. above its b. pt. at atmospheric pressure; that is, at a pressure of roughly 15 lb./sq. in. S. F. B.

1228. Process for Converting Petroleum Hydrocarbons. R. Cross. U.S.P. 1,819,542, 18.8.31. Appl., 17.3.26.

A process is claimed wherein the oil is raised to cracking temperature in a two-stage heating coil and cracking is effected in a reaction chamber. The vapours evolved are subjected to a refluxing action out of contact with raw feed, and the reflux condensate is recycled directly to the heating stage, receiving the most intense heat, and there mixed with the fresh feed. The vapours passing overhead from the reflux tower are condensed to form the cracked product (gasoline). R. P.

Hydrogenation.

1229. Reactivation of Catalyst used in Destructive Hydrogenation. Standard Oil Dev. Co. E.P. 352,850, 16.7.31. Appl., 20.10.30. Conv. (U.S.A.), 13.11.29.

In the hydrogenation of oils with catalytic agents there is a steady decrease in catalytic activity which is not due to sulphur poisoning. It has been discovered that the catalyst can be reactivated by treatment with hydrogen either at ordinary or at elevated temperatures and pressures.

If the gases from a hydrogenation retort be used, it is preferable that they should be scrubbed to free them from hydrocarbons and hydrogen sulphide; but if an unscrubbed gas be used it is desirable that comparatively pure hydrogen be passed over the catalyst before it is returned to the process.

The catalysts referred to consist of the oxide or sulphide of chromium, molybdenum, tungsten, or similar high atomic weight material, either alone or mixed. R. P.

1230. Catalytic Hydrogenation of Hydrocarbon Oils. R. P. Russell and M. W. Bozer, Assrs. of Standard Oil Dev. Co. E.P. 353,792, 30.7.31. Appl., 17.10.29.

When hydrogenating heavy hydrocarbon oils by passage through a heated zone packed with catalytic material with hydrogen under pressure, it is advantageous, when the activity of the catalyst has fallen beyond a satisfactory degree, to replace the heavy oil with a lighter oil, while still maintaining hydrogenating conditions, and then resuming the treatment of the heavy oil. The lighter oil should possess solvent properties so that it removes tarry material from the catalyst, and while doing so is hydrogenated. If solvents other than hydrocarbon oils are employed, it is generally necessary to lower the temperature, which increases the period required for cleaning the catalyst, while the use of hydrocarbons, such as spindle oils or kerosine, enables the hydrogenating conditions to be maintained while considerably improving the value of the solvent at the same time. S. F. B.

Chemistry of Petroleum.

1231. Heat Treatment of Hydrocarbons with Special Reference to Gaseous Hydrocarbons. A. E. Dunstan, E. N. Hague and R. V. Wheeler. *J.S.C.I.*, 1931, **50**, 313T-318T.

Modern developments have led to attention being given to the production of motor spirits of high anti-knock value from natural gases, etc. This paper deals with this question from the point of view of heat treatment of gaseous hydrocarbons. An interesting historical survey with many references precedes a study of the theoretical considerations involved.

The primary decomposition of a paraffin hydrocarbon indicates that the rupture of the chain can take place at any position, producing an olefine and the complementary lower paraffin, or at the limit, hydrogen. The absence of a C-C linking in methane causes it to differ from the carbon-chain paraffins and increases its stability. With the olefines, ethylene, propylene and the two unbranched butylenes, the principal primary reactions involve the formation of either the two- or four-carbon atom members (or both) of the olefine series, while under drastic conditions, secondary reactions occur with the decomposition of butylene to form butadiene and hydrogen in one case,

and propylene and methane in the other. A secondary change with propylene is the formation of methane and ethylene, probably by hydrogenation. $\Delta\beta$ -butylene was more stable than $\Delta\alpha$ -butylene.

The behaviour of all the paraffin hydrocarbons at high temperatures is similar, but below 725° C., when primary reactions are taking place, each hydrocarbon follows its own course. Between 700-750° C. (transition period) with all paraffins except methane, the production of ethylene reaches a maximum. The common source of the aromatic hydrocarbons produced above 750° C. is a C_2 unsaturated hydrocarbon and it is concluded that ethylene and not acetylene fulfils the necessary conditions. Ethylene readily polymerises to butylene, by dehydrogenation forms butadiene and the latter unites with ethylene forming cyclohexene which finally produces benzene. Aromatic hydrocarbons (mainly benzene) are produced from paraffin hydrocarbons by the secondary reactions above 725° C. production increasing with rise of temperature up to a maximum at a definite temperature, above which deposition of carbon occurs.

A table given for paraffins indicates that under optimum conditions methane requires a temperature of 1050° C. producing approximately 9% of liquids, while the hydrocarbons ethane to pentane require a temperature of 800-900° C. producing 22-27% of aromatic liquids. Hexane gives a yield of 34% at 800-850° C. The liquids produced from methane consist of 80% benzene, but the liquids from the other paraffins contain 50% of "spirit" boiling below 170° C., which is chiefly benzene, other constituents being, toluene, styrene, xylene and naphthalene. With olefines, as with paraffins, secondary reactions occur which lead to the production of aromatic hydrocarbons. A table given illustrates that between 700-800° C., the conversion is equivalent to 36-40% of the olefine passed, the liquid produced containing 50-60% distilling below 170° C. W. A. W.

1232. Formation of Oil from Dilute Acetylene. D. Binnie. *J.S.C.I.*, 1931, **50**, 297T-299T.

When mixtures of 4% dry acetylene and 96% hydrogen were passed through a silica tube at 600° C. the chief product was ethylene with some methane, but no ethane or oil were formed. Metallic catalysts (in the form of nickel carbonyl) were then introduced, but owing to excessive hydrogenation to ethylene and ethane, a mixture of 4% dry acetylene, 20% hydrogen and 76% nitrogen was used at a temperature of 400° C., this producing about 40% of oil (based on ingoing acetylene). Diminution in the activity of the catalyst was overcome by introducing the nickel carbonyl in the form of "catalytic" gas into the inlet gas, in such quantity that it did not exceed 4% of the total furnace gas. The exit gas was cooled in liquid air, and from the product, ethylene, ethane, etc., were boiled off leaving an oil. Yields up to 60% were obtained. Nickel carbonyl can be replaced by its components, the furnace being coated with nickel, and the carbon-monoxide being introduced with the inlet gas, thus 2% of carbon-monoxide added in place of the carbonyl gave oil yields of 50-55%, but when the carbon-monoxide was cut off the oil yield dropped to 6%.

For best yields of oil a 4% acetylene mixture requires 12% of hydrogen and the carbon-monoxide content need not exceed 2%. Smoother running experiments are obtained in tin or nickel rather than silica tubes.

The oil produced from silica and nickel tubes had a S.G. of 0·69 and boiled from room temperature to 85° C.; it is essentially olefinic. Oil obtained from an iron tube had a S.G. of about 0·73 and boiled between room temperature and 200° C. Five tables of results of different experiments and conditions are given. W. A. W.

1233. Generalised Thermodynamic Properties of Higher Hydrocarbon Vapours. J. Q. Cope, W. K. Lewis and H. C. Weber. *Ind. Eng. Chem.*, 1931, **23**, 887-892.

All the available direct experimental determinations of deviations from the gas laws for the saturated vapours of hydrocarbons possessing more than two carbon atoms per molecule when plotted against the ratio of reduced pressure to reduce temperature fall on a single curve, the maximum deviation being 3%.

Analysis of the data leads to the conclusion that, up to moderature pressures, the deviations from the gas laws for hydrocarbons having more than three carbon atoms per molecule are roughly the same for equal values of reduced pressure and temperature whether they are saturated or not. Graphically these relations are valid for a value of $\frac{RT}{P_cV}$ = about 4 but not above.

As a result it is possible to estimate the vapour volume and density of a hydrocarbon under any required temperature and pressure from a knowledge of molecular weight, critical pressure and the temperature. These graphical relationships may be expressed by approximate algebraic forms corrected by graphical functions obtained from the original data, from which, by graphical integration, the internal energy and total heat of hydrocarbon vapours as functions of temperature and pressure are obtained. It is thus possible to determine the heat consumption and energy changes of various processes. Furthermore if the additivity of volumes and total heat when vapours are mixed at constant pressure and temperature are assumed, the volumetric and thermal relations of vapour mixture can be estimated.

S. F. B.

1234. Velocity Constants for the Thermal Dissociation of Ethane and Propane. L. F. Marek and W. B. McCluer. *Ind. Eng. Chem.*, 1931, **23**, 878–881.

The authors have determined the velocity constants for the dissociation of ethane and propane and correlated these data with values calculated from the published data on the pyrolysis of these hydrocarbons.

The pure gases (ethane from the hydrogenation of ethylene over a nickel catalyst at 160° C., and propane from Pyrofax by fractionation) were quickly heated to reaction temperatures and passed through a copper reactor at carefully controlled temperatures with short contact periods. The extent of cracking was determined by the difference in composition of the preheater and reaction coil samples, and values for the velocity constants were calculated from the general equation for unimolecular homogeneous gas reactions. The values found for the velocity constant for the thermal dissociation of ethane and propane and the hydrogenation of ethylene in the temperature range 600–700° C. are represented by the respective equations :—

$$\log k_{C_2H_6} = 15{\cdot}12 - \frac{15{,}970}{T}$$

$$\log k_{C_3H_8} = 13{\cdot}44 - \frac{13{,}500}{T}$$

and

$$\log R^1 = 8{\cdot}79 - \frac{9140}{T}$$

With short times of contact and at temperatures below 700° C., ethane decomposes predominately to ethylene and hydrogen, and secondary reactions, polymerisation and demethanesation of ethylene, are negligible. On the other hand, the principal reaction involved in the thermal decomposition of propane are dehydrogenation and demethanesation, the rates of the two reactions being approximately the same.

S. F. B.

1235. Detection and Separation of Hydrocarbons with Branched Chains from Natural or Artificial Mixtures of Hydrocarbons. A. Schaarschmidt. *Chem. Ztg.*, 1931, **55**, 424.

The olefines and aromatic compounds present in hydrocarbon mixtures are easily determined and separated, and in the residual mixture of saturated aliphatic and alicyclic hydrocarbons, the latter may be determined with moderate accuracy, although not isolated, by means of the aniline point, in accordance with Tizard and Marshall's method. It is now found that, with the more simple mixtures obtained from natural or synthetic hydrocarbons, treatment with a metallic halide with a mobile halogen atom, such as antimony pentachloride, renders it possible to detect and separate saturated aliphatic and alicyclic hydrocarbons containing trebly-linked carbon atoms; the complex compounds formed are insoluble in excess of the hydrocarbon mixture, and form characteristic greenish-blue or violet products with water or alcohol. Normal hydrocarbons and those containing quaternary-linked carbon atoms remain behind. This method has been applied to different fractions obtained from synthetic and natural hydrocarbons, it being found, for example, that benzine fractions with a b. pt. of about 100° C. consist mostly of aliphatic and alicyclic hydrocarbons with branched chains. W. S. E. C.

Analysis and Testing.

1236. Petroleum Asphalt. H. Pöll. *Erdöl und Teer.* 1931, **7**, 350–352, 366–371.

A large amount of work has previously been reported, having as its object the separation of asphalt into its component groups. The methods depend upon the selective action of solvents and absorbing media. A description and criticism of the methods of Richarson, Holde, Simmersbach, Marcusson and Eickmann, Nastjukow, Marcusson and Picard, and Suida and Kamptner is given. From the results of consideration and trial of these methods the author proposes the following process:—

The asphalt is shaken in the cold in a closed rolling-machine with ten times its amount of petroleum ether boiling up to 50° C. The so-called asphaltenes are filtered off after 24 hours, and the solution treated with two or three times the amount (on the asphalt) of bleaching earth, which removes the resins. (If the earth is not dried the oil is not extracted, but only the resins.) The filtrate is again extracted with earth and the earth washed with petroleum ether. The united filtrates are evaporated to give the "petroleum oil fraction."

The "petroleum resins" are obtained by extracting the earth with chloroform.

The precipitated asphaltenes are dissolved in chloroform and shaken in the cold with the earth (which still contains asphalt-resins) until the chloroform is water-bright. The filtered-off earth is then extracted with pyridine in the cold, yielding, after suitable treatment, the "asphalt resins." Finally, the hard asphalt is obtained by extraction with a 1 : 1 mixture of pyridine and carbon disulphide.

The advantages claimed for this method are—a much sharper separation of the asphalt into constituent groups, independence of benzine for the separation excepting for the fairly well-defined petroleum ether which boils up to 50° C. and contains no aromatic or unsaturated hydrocarbons. The method is applicable to all heavy petroleum products from spindle oils to superheated cylinder oils, as well as to asphalt.

Comparative tables are given showing the results of separation of an asphalt by the various methods described and elementary analyses and properties of the separated constituents. E. B. E.

1237. Investigation into the Nature and Properties of Coal Tar. E. V. Evans and H. Pickard. *Fuel*, 1931, **10**, 352–369.

Part I.—Relationship between Temperature and Consistency of Coal Tar and Other Bituminous Binders.—The available information in the literature is briefly reviewed. The general expression $C.T^n = a$ shows the relationship between temperature and consistency (C) of a tar, where c may be expressed in terms of the Hutchinson instrument, the tar viscometer or the Redwood No. II. viscometer. From this it follows that—

$$n = \frac{\log C_1 - \log C_2}{\log T_2 - \log T_1}$$

It is therefore sufficient to determine the consistency at two temperatures only to ascertain the consistency at any other temperature. Chemical constitution has an appreciable effect on consistency and the values of "n" and "a" were determined for a synthetic tar and also for mixtures of it with varying amounts of benzene, phenol, aniline, α naphthylamine, naphthalene or β naphthol. A chart shows the logarithmic temperature/consistency relationship for all the tars examined. Apparently there is a common point of intersection, indicating that theoretically at one temperature the consistencies of all the different tars are equal.

Part II.—Preparation of Asphaltic-like Substances from Coal-Tar.—A simple viscometer (called the Metro Trough viscometer), designed to eliminate defects in existing types, is briefly described and illustrated. A constant volume of material is placed in the well of the instrument and after the required temperature is reached the time taken for the material to flow between two fixed marks when the trough is inverted to a definite angle is noted. By means of this instrument the relative differences betwen tar and asphaltic bitumen were studied. It was found that taking asphaltic bitumens of the same origin, the focus of the curves lies higher up the temperature scale than the corresponding point for tar. In an attempt to make tar more like asphaltic bitumen as regards temperature/consistency relationship, it was found that the addition of finely-divided lampblack to tar caused an upward movement of the curve parallel to that of the original tar, moving the position of the focus. The extent of the parallel movement is a function of the amount of dispersoid present and also the size of the particles dispersed. A method is therefore available for estimating and measuring the influence of free carbon on tar and the effect of fillers on consistency. A tar may be made less susceptible to the influence of temperature by dispersing in it a suitable quantity of finely divided matter.

Part III.—Consistency of Pitch-like Substances.—An instrument (called the Metro Plastimeter) has been devised to measure the rate of flow of pitch and similar substances under heavy loads. A sphere of steel attached to a supporting rod is forced by the application of a suitable weight into a mould of the material under examination, the rate of penetration being measured by observation of the depth of penetration at various times. Results obtained by this apparatus on samples of pitch and asphaltic bitumens are given. It is concluded that the pitch exhibits the characteristics of a viscous fluid, but asphaltic bitumen shows anomalous consistency effects in its velocity/load relationship, and belongs to the class of plastic solids. By dispersing coal in tar, however, the resulting system possesses the physical characteristics of asphaltic bitumen rather than the fluid properties of coal tar. C. C.

1238 Criteria of the Degree of Refining of Lubricating and Insulating Oils by the "Sulphuric Acid Absorption Curve." G. Tœnnies. *Petr. Zeit.*, 1931, **27**, 527–34.

A process is described for the quantitative determination of the content of constituents in lubricating oils which are unstable towards sulphuric acid concentrations lying between 90 and 100%. The percentage of acid is plotted against the percentage absorption (percentage of oil absorbed by the acid) for a number of lubricating oils and the curves obtained are smooth and have an individual and characteristic form for any oil. A comparison of the sulphuric acid absorption curves for a number of different oils with the stability to oxidation shows that certain relations exist between the stability to oxidation and tendency to sludge formation of an oil and the position of its absorption curve.

Various curves are given for different types of lubricating oils. W. S. E. C.

1239. Testing Rates of Solution of Oil and Gas. W. N. Lacey. *Oil and Gas J.*, 1931, **30** (8), 15; and **30** (9), 68.

In consideration of the fundamental principles of the repressuring process, the two important factors are (1) the maximum quantity of gas which can be introduced into a formation (which is related to the total solubility under given conditions), and (2) the time taken to get a given quantity of gas into solution. The present investigation (Project No. 37 of the A.P.I.) attacks the second problem.

The absorption apparatus used consists of a cylinder 4 in. high by 1·4 in. in diameter, carrying 80 ml. of liquid (generally crude oil). Gas is continuously introduced into the air space at constant pressure and the rate of diffusion of the gas into the oil measured from a consideration of the volume of gas introduced. The latter is determined by the decrease in pressure of the main body of the gas maintained in the reservoir. Absorption at first is very rapid until the surface is saturated, diffusion then taking place more slowly. The apparatus was maintained at 86° F., the gas used being natural gas filtered through activated charcoal and containing 98% of methane. Complete saturation point was determined by violent agitation of the absorption cell.

The fundamental equation of rate of diffusion is obtained, it being shown that the quantity of gas passing the surface in unit time is equal to a constant multiplied by the cross section of the cell and also by the concentration gradient. The fraction of saturation attained (i.e., the quantity of gas absorbed divided by the quantity required for total saturation) is shown to be a function of the depth of the cell and of the time. In cases where the gas has not yet reached the bottom of the cell the quantity of gas going into solution is proportional to the square root of the time, whereas after this has occurred it is an exponential function of the time. A diagram showing the actual distribution of the molecules in the liquid indicates that 50% saturation of the liquid is not reached by the time the first molecules reach the bottom of the cell, this fraction of saturation not being attained until the concentration at the bottom is 22%.

Corrections have to be applied for the amount of gas going into solution immediately on introduction before the pressure is held constant, as well as for the increase in volume of the liquid due to the presence of gas.

A series of tests (full details of which are given) were carried out on various petroleum hydrocarbons and crude oil fractions, using methane as the gas.

Tests at various temperatures indicated that the rate of diffusion is greater at the higher temperature, in spite of the fact that the concentration gradient is smaller owing to decreased solubility at higher temperatures. In the

presence of sand, the actual rate of diffusion is much slower, although when calculated on a basis of effective oil surface (total pore space of sand divided by depth of layer) the figure is only slightly lower than usual.

Ethane in cyclohexane showed a much higher rate of diffusion than methane, owing to the greater solubility.

It was found in general that the rate of solution of methane was in proportion to the A.P.I. gravity of the liquid, though there is no definite theoretical connection.

Further work has been carried out on the total volume of natural gas which can be dissolved under pressure in different types of crudes. C. L. G.

Anti-Detonation.

1240. Effect of Humidity and Air Temperature on Octane Numbers of Secondary Detonation Standards. D. B. Brooks, N. R. White and G. C. Rodgers. *J.S.A.E.*, 1931, **29**, 45–51.

Tests have been carried out at the Bureau of Standards to determine the influence of air-conditions on knock-rating results. Using the C.F.R. engine with an apparatus for controlling the humidity and temperature of the intake-air, the octane numbers of the following secondary standards were determined: 1. (*a*) Michigan gasoline plus tetraethyl lead; (*b*) Michigan gasoline plus benzole. 2. (*a*) Cabin Creek gasoline plus tetraethyl lead; (*b*) Cabin Creek gasoline plus benzole. 3. Heptane plus benzene. 4. Texas spirit plus Californian aviation gasoline.

All the ratings were made directly against octane-heptane except in the case of No. 2, which was rated against No. 1, this having previously been calibrated against octane-heptane.

In the case of the last two standards the temperature as well as the humidity was varied, air-intake temperature of 40° and 60° C. being used. The humidity variations employed were approximately 6 mm. and 45 mm. of mercury.

In the case of the first three scales there is very little effect of humidity on the relations of the secondary to the primary scale. With the last scale, however, the effect is quite marked, amounting to about six units at the lower end of the scale and about two units at the upper. An increase of about 40 mm. in the humidity increases the anti-knock value of both Texas and California spirit, the former more so than the latter.

Air-temperature has an effect which is due to the increase in anti-knock value of benzole at higher temperatures. Lead is practically unaffected in this respect. The California gasoline in the last standard is affected much in the same way as benzole by air temperature, while there is little effect on the detonation of Texas spirit. It is, therefore, necessary to study the effect of atmospheric conditions on materials intended for use as secondary standards. E. B. E.

1241. Jacket and Cylinder-Head Temperature Effects upon Relative Knock-Ratings. G. Edgar. *J.S.A.E.*, 1931, **29**, 52–56.

The effects of varying jacket and cylinder-head temperatures, spark advance and compression ratio on the knock ratings of various fuels have been investigated. Two series of experiments were conducted. In the first series five spirits, including a benzol blend, a leaded gasoline and a cracked gasoline were matched by additions of lead or benzol to a straight-run Pennsylvania gasoline. Various compression ratios and jacket temperatures of 212° F.

and 350° F. were used, the spark being adjusted in each case for maximum power. The cylinder-head temperatures were measured with a thermocouple. In the second series the amounts of lead required in a Mid-Continent gasoline to match the same gasoline containing various percentages of benzene were determined at jacket temperatures (212, 275 and 350° F.) and at different compression ratios. The spark advance was maintained at 15° C.

The general results show that at higher jacket temperatures less lead is required to match a given amount of benzol in the gasoline. In the second series, at higher compressions lead becomes more effective with respect to benzene. Series one shows no definite change in this way. The difference in results is attributed to the position of the spark—adjusted for maximum power in one case and kept at constant setting in the other. This was supported by some further experiments. It is thought, although not conclusively proved, that all the results may be functions of cylinder-head temperature alone. E. B. E.

1242. Effect of Oil Consumption and Temperature on Octane Number Ratings. H. R. Stacey. *J.S.A.E.*, 1931, **29**, 57–58.

Many users of knock-testing engines have found the results obtained subject to variation. Part of this variation is caused by the oil itself, and this effect has been investigated. An Ethyl Gasoline Corporation engine was used, fitted with a thermocouple to measure the temperature of the oil in the sump. The two oils used were Pennsylvanian and Coastal, with Saybolt viscosities of 62 secs. at 210° F.

Using Pennsylvanian oil at 110° F. in the crankcase and a close-fitting piston with two oil-rings to keep the consumption down to ¼%, a Californian gasoline was matched by a poor anti-knock spirit to which 1·8 c.c. tetra-ethyl lead or 34% benzol had been added. Three spirits were thus obtained with an octane number of 67·6 under these conditions. Tests were then run to determine the octane numbers of the fuels at oil sump temperatures of 110° and 185° F. with both Pennsylvanian and Coastal oils. Experiments were also run with 4% and 8% respectively of oils dissolved in the fuel.

With low oil consumptions changing the oil made no difference to the results. Both oils, when added to the fuels, lowered the octane numbers at both temperatures, having slightly more effect on the lead and benzol blends. In the case of the 8% blends the Pennsylvanian oil had a greater deleterious action than the Coastal.

There are two effects coming into play in these experiments, (1) partial admixture of oil with the charge by leakage past the piston and (2) differences in rate of cooling due to different amounts of oil flowing between piston and cylinder wall. By using a loose-fitting piston the consumption of oil was increased from ¼ to 2%. The octane number dropped in each case from 67·6 to 66·9, and the engine became noticeably erratic with the higher oil consumption.

The secondary standards used were rated against iso-octane-heptane direct at oil temperatures of 110° and 185° F. No difference was observed. A 50% benzol blend, however, dropped half an octane number at the higher temperature. The equivalent lead blend is not affected.

It is concluded that errors in rating commercial fuels due to differences in oil consumption or oil temperature may amount to about one octane number for each factor. All knock-testing engines should be designed for as small an oil consumption as is consistent with safety and the oil temperature kept as low and constant as possible. The use of a universal standard is also recommended. E. B. E.

1243. Influence of Carburettor Setting and Spark Timing on Knock Ratings. J. M. Campbell, W. G. Lovell and T. A. Boyd. *J.S.A.E.*, 1931, **29**, 129–133.

The engine used was the Horning variable compression engine running at a constant speed of 600 r.p.m., with a jacket water temperature of 212° F. and a bouncing-pin indicator. Intake-opening was constant in the absence of a throttle. Mixture strength was altered by adjusting the level of float bowls feeding fixed jets. Spark timing was followed by a neon-tube and three pairs of fuels were compared under different combinations of carburettor setting and spark timing.

(1) *40% of Benzene in Gasoline and varying amounts of Tetraethyl in Gasoline.*—With these fuels, as the timing of the spark is advanced, two important changes take place in their relative knock ratings:—(1) Greater concentrations of lead tetraethyl are necessary to match a given concentration of benzene in the same gasoline; (2) There is an increase in the magnitude of the influence of carburettor setting upon the relationship between the anti-knock effect of lead tetraethyl and of benzene respectively.

Further graphical analysis shows that, at spark timings *before* that for maximum power, the effect of spark timing on the comparison of two such fuels is more pronounced than at spark timings *after* that for maximum power.

(2) *A Straight-run Gasoline* (*AAA*-1) *compared with Benzene or with Iso-Octane in Heptane.*—Comparisons were made with carburettor settings giving maximum knock. The results obtained showed that the concentrations of benzene or of iso-octane in heptane found to equal gasoline AAA-1 depended somewhat upon the timing of the spark when comparison is made; as spark timing was advanced, the knock intensity increased with all three fuels and more important still, the rate of increase of knock intensity with respect to spark advance was greater with gasoline AAA-1 than with mixtures of benzene and heptane.

(3) *Benzene in Heptane compared with Iso-Octane in Heptane.*—The results obtained indicate that the knock rating of one reference fuel with respect to another appears to depend upon both the spark timing and upon the carburettor setting at which the comparison is made.

The net result of the work suggests that in making knock measurements, the necessary comparisons of the fuels should be made at carburettor setting for maximum power and at spark timing for maximum power. C. I. K.

1244. Effect of Sound Intensity on Knock Ratings. H. F. Huf, J. R. Sabina and J. B. Hill. *J.S.A.E.*, 1931, **29**, 134–6.

Hitherto, doubt has existed in discussions of the possibility of one of two fuels being able to cause destructive detonation before the other, even though the two fuels cause incipient detonation under identical conditions. In order to clarify the situation, a knock-audiometer (a microphone, a vacuum-tube amplifying and filtering circuit and a highly damped milliammeter) was developed and is described.

Widely varying fuels were compared with mixtures of normal heptane and iso-octane at knock intensities ranging from incipient knock to a knock severe enough to cause pre-ignition. The engine employed was the L-head C.F.R. engine.

The conclusion drawn is that the degree of detonation at which a comparison of fuels is made does not affect appreciably the knock ratings of the fuels in the engine used, provided the fuel is matched with a sub-standard reference fuel of approximately the same knock rating as the sample.

The audiometer has been found very satisfactory for determination of knock ratings and should be of wider value since it eliminates the human element which is affected by ear fatigue. C. I. K.

1245. Bouncing-Pin *versus* Throttle Audibility. N. MacCoull. *J.S.A.E.*, 1931, **29**, 137–8.

The standard procedure for the Ethyl Gasoline Corpn. engine was used in the determination of the percentage of benzol to be added to a commercial gasoline to equal the same anti-knock values of 1, 2, 3 and 4 c.c. of lead tetraethyl per gal. of the same gasoline. Readings obtained with both the bouncing-pin knock-meter and the throttle-audibility methods agreed satisfactorily, the average difference being only ¼% of benzole.

Another series of tests consisted of matching varying amounts of lead in a reference gasoline by adding 0·2 c.c. increments of lead to the same reference fuel. Results are obtained by the bouncing-pin method on the S. 30 engine and the throttle-audibility method on the L-head C.F.R. Engine. No appreciable advantage existed in the accuracy of one method compared with the other. These findings receive support from the results of Huf, Sabina and Hill (see preceding abstract) since light knock obtains with the throttle-audibility method and the bouncing-pin requires heavy knock.

C. I. K.

1246. Time-Lag in Detonation Determinations. N. MacCoull. *J.S.A.E.*, 1931, **29**, 139–40.

Particularly when two fuels of equal knock-rating, but having widely different compositions (a benzol blend and a leaded gasoline) are fed into a test engine through a double float carburettor, one can observe either a disappearance of knock, an increase or decrease of knock immediately after switching over from one fuel to the other. The time required for a uniform knock value to be reached after switching over is called the time-lag. This period may be 7 min. in duration, so that knock readings taken within that period can lead to erroneous results.

Experiments showed that variations of piston head temperature occurred during the period immediately subsequent to switching over and that this temperature was substantially the same for all fuels.

Provisions made to produce rapidly dry air-fuel mixtures after carburetion reduced this time-lag (raising the jacket temperature to 300° F. or better, steam-jacketing the pipe between the carburettor and cylinder block).

C. I. K.

1247. Tentative Recommended Practice for Conducting Anti-Knock Tests. Detonation Sub-Committee of the C.F.R. Committee. *J.S.A.E.*, 1931, **29**, 164–5; 168.

The procedure recommended is the one of several available methods, most likely to give reproducible results, but its use is not advocated for aviation fuels which will be provided for in a later publication.

The engine to be used is the C.F.R. Engine frequently referred to as the "Horning Engine." The knock measuring device is a bouncing-pin and a gas evolution burette (standard equipment) or a knock meter (at purchaser's request). Knock ratings are to be expressed as "Octane Numbers" and a scale extending beyond "100 Octane Number" is suggested (100 plus the percentage of benzene in iso-octane mixtures). The suppliers of octane and heptane are quoted but two cheaper secondary standards having octane numbers of 50 and 68 respectively are available. The details given deal with the apparatus, foundation for it, engine control, standard adjustments,

reference fuels, knock intensity, adjustment of mixture-ratio, anti-knock determination, maintenance, and an appendix on engine and bouncing pin adjustment. This last item includes remedies for the difficulties and abnormalties encountered with such a method of test. C. I. K.

1248. Detonation, Spark-Plug Position and Engine Speed. R. O. King and H. Moss. *Engineering*. 1931, **132**, 177.

The authors have investigated the influence of sparking-plug position on the H.U.C.R. of doped and undoped fuels in a Ricardo E35 variable-compression engine. This is an overhead-valve engine with two large inlet valves at one side of the combustion space and a group of three smaller exhaust valves at the opposite side. Four sparking plug positions are provided, any two of which are used simultaneously, the four being spaced symmetrically round the cylindrical combustion space. The compression ratio was determined for each fuel at which slight audible detonation was obtained at full throttle, 60° F. jacket temperature, and mixture for maximum detonation.

In a preliminary series of experiments, a comparison was made between the H.U.C.R. and B.M.E.P. obtained using two plugs on the exhaust valve side with those obtained using two diametrically opposed plugs, one on the inlet and one on the exhaust side. The three fuels used were: (1) a commercial brand of aviation spirit (petrol A) containing 57% paraffins, 35% naphthenes, and 8% aromatics by weight; (2) petrol A, 2 parts, benzole 1 part; (3) petrol A plus 4 c.c. ethyl fluid per gal. The engine was run at 1500 r.p.m. with unheated air at ignition timings from 25°–45° advance. The H.U.C.R. results were practically unaffected by the change in plug positions. With petrol A alone, the B.M.E.P. was higher over the whole ignition range when the diametrically opposite pair of plugs was used. For the blended fuels the curves of B.M.E.P. against ignition advance intersect near the advance for maximum power (35°), giving higher power for the diametrically opposed plugs at greater advance and lower powers when the ignition was retarded.

The next series of experiments was carried out to determine the effect of reversing the direction of flame movement with respect to the inlet and exhaust valves. This series was carried out at fixed ignition advance (30°), but at two speeds, since increased speed would increase the temperature difference between the inlet and exhaust valves. Petrol A alone and various blends with benzole and with ethyl fluid were investigated. The inlet air was unheated in one set of experiments and at 50° C. in the other set. In all cases a drop in H.U.C.R. was experienced when the flame movement was directed towards the hotter exhaust valves. With benzole mixtures, indeed, the difference tended to equal the improvement in H.U.C.R. due to the increased engine speed, particularly at the higher induction temperatures. Thus the H.U.C.R. at 1500 r.p.m. tended to equal that at 900 r.p.m. when the flame movement was towards the exhaust valves. This temperature effect at high induction temperatures did not appear for ethyl fluid blends. It was shown, however, by undoped fuels containing higher concentrations of naphthenes than fuel A. These results are of particular interest with regard to supercharged engines for which the induction temperatures may easily exceed 50° C. For such engines the H.U.C.R. will be very sensitive to the direction of flame movement and the nature of the fuel. Fuels containing a high percentage of aromatics or naphthenes may entirely lose the beneficial effect on H.U.C.R. of increased engine speed. Ethyl doped spirits will not, however, suffer from this defect.

It was observed during the preceding experiments with fixed ignition timing that the higher compression ratios permissible when the flame movement was towards the cooler inlet valves did not produce the expected increase

in power. In order to get the full benefit a greater ignition advance was required. This supports the view that combustion is more rapid when directed towards the hot exhaust valves. Similarly, when the compression ratio was fixed, as in practice, the power was independent of the direction of flame movement provided the ignition was slightly advanced when firing from exhaust to inlet side.

The authors conclude that the only advantage of the one direction over the other in practice depends on the comparative ability of exhaust valves and sparking plugs to withstand heating. Plugs placed near exhaust valves tend to overheat in extreme conditions, whereas ignition from the inlet valve side may overheat the exhaust valve region where combustion is completed. The ideal arrangement for the E35 engine is dual ignition from inlet and exhaust valve sides simultaneously. G. B. M.

Engines.

1249. Double-Acting Diesel Engine Development. L. R. Ford. *Mar. Eng.*, 1931, **54**, 347–9.

The mechanical complications of the 4-str. cycle, more pronounced when both ends of the cylinder were used for combustion, have been mostly eliminated by the use of the 2-str. cycle. The assumed difficulty of obtaining good combustion in the lower end of the cylinder has proved to be less in magnitude than expected. The provision of good distribution of fuel in the lower combustion chamber, in which the piston is an obstruction, has been effected in four-stroke cycle engines by having a separate combustion chamber cast on one side of the lower cylinder head, and in the two-stroke engines by using two or more injection valves—the piston passing coaxially through the lower chamber. The leakage round the piston rod operating in an atmosphere of flaming gas at high pressure has been overcome. In the Burmeister and Wain engine the piston was cooled with oil passing through an annular space in the rod, and also snap rings (with an "inward" tendency) were provided in the stuffing box.

Airless injection of the fuel has been successfully applied, with a result that cylinder size has increased. Owing to the limitation by the dense air on its penetration of oil spray to about 7 to 8 in., proper distribution is obtained by multiple oil sprays.

Piston troubles have been minimised by cooling with water or oil passed through the piston rod, and this cooling has been carried out in different manners, though the structural features of double-acting pistons are substantially well standardised in principle.

Piston rod failures, now not common, were mainly due to the location of, and the manner in which, the inlet and outlet pipes for the cooling medium were fixed to the piston rod.

There is a definite trend towards double-acting two-stroke engines with mechanical injection of the fuel. C. I. K.

1250. Compression Ignition Engines. (A Return to Fundamentals in a Comparison with the Petrol Engine.) H. T. Tizard. *Aircraft Eng.*, 1931, **3**, 185–186, 196.

The fundamental differences between petrol and compression igni ion engines are discussed. For engines of either class, high efficiency demands high maximum pressures, but considerations of lightness, detonation (on

petrol engines) and pre-ignition (on compression ignition engines) limit these pressures. Whereas, in the petrol engine at least 80% of the air must be burnt, in the compression ignition engine, it has not yet been found possible to burn effectively over 80% of the air. High thermal efficiency can only be obtained if weak mixtures can be burnt effectively, while maximum power is limited by the amount of air that can be burnt. On the basis of certain stated assumptions, the theoretical thermal efficiency of petrol engines can be represented by the expression :—

$$\delta = 1 - \left(\frac{1}{r}\right)^{0.287} \quad \text{where } r = \text{compression ratio.}$$

Assuming a constant pressure cycle, for the compression ignition engine, when combustion is complete the final expansion ratio is given by :—

$$r_e = \frac{Tc}{(1+n)\,T_e} \times r_c \quad \text{(allowing for increase in volume due to combustion),}$$

where T_c = compression temperature, T_e = maximum explosion temperature $(1 + n)$ = ratio of number of molecules after combustion to number before combustion. A table gives results of calculations of limiting efficiencies of compression ignition engines over maximum pressures ranging from 324 to 2040 lb./sq. in., the efficiencies varying from 0·37 to 0·59. The method of calculation is indicated.

By means of curves showing the relation between theoretical efficiencies and maximum pressures for the two types of engine (*i.e.*, constant volume and constant pressure), the latter are compared. Similarly the corresponding compression ratios are plotted against maximum pressure. The difference in efficiency gets less as the maximum pressure is raised, the constant pressure engine having a higher efficiency than the constant volume type at a given maximum pressure. The author considers 1500 lb. as the limiting pressure in practice for the constant pressure engine, but actual pressures usually run from 800 to 1000 lb., corresponding to theoretical efficiencies of 0·5 and 0·52. There is little advantage in increasing the compression ratio above 13 to 1 for maximum pressures of this order.

The difficulties of burning the whole of the air in a compression ignition engine are discussed from a theoretical point of view, and it is suggested that the aim should be to inject the fuel at right angles to the direction of motion of the pistons to ensure that the fuel is mixed with the unburnt air as it enters the cylinder.

Results from boosting compression ignition engines are disappointing, for raising the initial pressure not only lowers the efficiency but reduces still further the proportion of air that can be burnt; the efficiency should, however, remain high compared with that of the petrol engine, and there are other advantages in theory. Experimental work is required on the effect of increasing the initial pressure; supercharging will almost certainly be required for altitude conditions.

Two-stroke operation is more feasible in a compression ignition engine. It offers a possibility of reducing the weight/power ratio to a figure comparable with that of the petrol engine.

The author considers it unlikely that the power/weight ratio of normally operated 4-stroke compression engines can be reduced sufficiently to compete successfully with petrol engines for any length of flight that is reasonable under commercial conditions. C. C.

1251. Compression-Ignition Engines. P. A. Poppe and Dennis Brothers, Ltd. E.P., 352,227, 9.7.31. Appl., 26.6.30.

By improved arrangements of air supply in C.-I. engines having ante-chambers into which the fuel is injected, very complete and rapid combustion is obtained, so that the engine will operate at relatively high speeds. The ante-chamber of venturi shape, fully open at the piston end, is provided with an air-inlet valve at the point most remote from the piston. The scavenging of the remote end of this chamber is effected at each suction stroke and the fuel is injected into relatively pure air. A high volumetric efficiency is obtained, and the engine can be run at relatively high speeds to produce higher power, owing to the complete combustion of the fuel. J. M. F.

1252. Internal Combustion Engines Operating with Liquid Fuel Injection. K. J. E. Hesselman. E.P., 352,794, 16.7.31. Appl., 2.9.30.

In I. C. engines with liquid fuel injection and external ignition, such as sparking plugs, and the air mass rotating, particles of the fuel which strike the wall of the recess, housing the plug, are thrown into the recess and may cause ignition failure.

By chamfering the edge of this recess on the side farthest from the igniter, in the direction of rotation of the air mass, particles striking this wall are deflected towards the interior of the combustion space, and not against the igniter. J. M. F.

1253. Supplying Liquid Fuel to the Cylinders of Oil Engines. Packard Motor Car Co. E.P., 352,983, 13.7.31. Appl., 11.3.30.

The invention relates to a method of minimising the amount of air, trapped in the fuel, fed to the pumps of solid injection oil engines.

The fuel is circulated, from a supply tank, under a low pressure through a manifold to and from chambers from which the fuel to be injected is withdrawn. The fuel is always fed upwards through the manifold, in which there are no pockets to entrap air. The amount of fuel circulated in a given time is about six times that actually injected into the cylinders. Thus any air that is in fuel does not reach the injection nozzles, but is swept through the manifold and back to the supply tank where it can separate from the fuel. J. M. F.

1254. Pre-Combustion Chambers of C. I. Oil Engines. J. I. Thornycroft & Co. and V. G. Barford. E.P., 353,149, 23.7.31. Appl., 22.5.30.

The patent describes a form of pre-combustion chamber which provides better mixing of the air and fuel, so producing more efficient combustion than usual. This chamber is situated at the side of the cylinder, and is connected to its upper end by a laterally arranged port, such that it imparts a swirling motion to the air as the piston forces it in. This gives a more intimate mixture of the fuel and air, the fuel being sprayed from the top or bottom of the pre-combustion chamber. J. M. F.

ABSTRACTS.

Geology.

1255. Petroliferous Region of Gura-Ocnitzei. E. Mircea. *Ann. des Mines Roumanie*, 1931, **14**, 297–303.

The Gura-Ocnitzei region may be divided into two parts. The old field extending to the Cezeanal Valley on the east is a continuation of the Diaper fold from Moreni. The south flank is now the more important, whereas the north flank has been largely drilled up. Production is mainly from the Dacic.

The new field is on an anticline whose axis is the continuation to the east of the Moreni–Gura-Ocnitzei line of salt. The northern flank is faulted and the most productive zone is in the Maeotic I north of the fault. On the south flank the production is from Maeotic III between two faults near the crest of the fold. The Dacic is unproductive.

South of these faults the area appears to be the normal continuation of the south flank of the old field. The thickness and lithology of the Maeotic are generally similar to the conditions at Moreni. The paper gives drilling and production data, and contains a structural map of the area as well as geological sections. P. A. S.

1256. Surface and Subsurface Character of Edwards Limestone. R. A. Jones. *Oil Weekly*, 1931, **62** (13), 18.

The Edwards Limestone—one of the most widely distributed formations in Texas—is of Lower Cretaceous or Comanche Age and is notable for its persistency in character over long distances. It is exposed continuously from the northern border of the State to the Rio Grande, and derives its name from wide occurrence in the Edwards plateau.

In the Balcones Fault region the limestone has been the reservoir rock of three great oilfields. It is usually crystalline, and characterised by the presence of nodular or concretionary chert and flint. There is little difference between the surface features of the limestone and those which cores and well-cuttings have shown it to possess in the sub-surface.

Chemical analyses of samples from the Salt Flat Field show it to be dolomitic in character (32% $MgCO_3$), and that one-fifth (20·6%) of the producing limestone is composed of organic material; insoluble material amounts to only 3·2%. G. S. S.

1257. Permo-Carboniferous Orogeny in South Central United States. W. A. J. M. Van Waterschoot van der Gracht. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 991–1057.

This paper deals with the tectonic movements which have affected the Palæozoic floor of the South Mid-Continent, a subject which is intimately connected with the general disposition of the oilfields. It is claimed that there are two independent fold systems; the Wichita and the Ouachita, of which the former is the earliest.

The Wichita fold system is exemplified in the Wichita Mountains, Criner Hills, Arbuckle Mountains and the Amarillo Hills. The general strike of the folding is east to west, and north-west to south-east, and is almost at right-angles to that of the later movement. The sediments of this fold system were laid down in a pre-Middle Devonian geosyncline, and in the highly compressed folding the predominant formations are marine limestones of Lower Palæozoic Age.

The Ouachita fold system is well developed in the Ouachita Mountains. The author claims, however, that the buried extensions of this system can be traced in a sinuously-curved line east of the Bend Arch and Llano Burnet Massif, south of which it bends again to the west and is exposed in the Marathon Mountains. East of the Ouachita Mountains the fold system disappears under a blanket of Cretaceous, but it is considered that it is directly connected under this cover with the Appalachian Mountain System.

The Ouachita fold system is claimed to be developed in a major geosyncline in which the maximum sedimentation was Mississippian and Early Pennsylvanian. Study of the Ouachita exposures leads the author to the conclusion that this area has undergone movements of an Alpine type, involving nappe structures and great overriding on to the foreland region to the north. The movements have been repeated over long intervals but the phase of maximum intensity culminated in great Pennsylvanian overthrusts and died out gradually in Permian times.

The Llano Burnet uplift is considered to represent a buttress around and against which the buried fold system bent; whilst in other parts, as in south-east Oklahoma and east-central Texas, the foreland is occupied by depressed basins of thick sedimentation. A generalised map, on which the main trend lines are placed, indicates the author's conclusions as to the general position of the main tectonic belts.

The paper contains an extensive account of the latest information on the facies of the formations and details of the structures in the area. These facts are interpreted by the writer along well-known lines set down by European geologists as confirmatory evidence of the great nappe movements. These, in his opinion, make the Ouachita fold system similar in structure to the Alpine system of Europe. V. C. I.

1258. Detailed Cross-Section from Yates Area, Pecos County, Texas into South-Eastern New Mexico. H. P. Bybee and Collaborators. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1087–1093.

This paper utilises the latest drilling information from over 3000 wells in the southern part of the Permian Basin to attempt an interpretation of the structure, and a detailed correlation across the basin. It is concluded that the Yates Sand of eastern Pecos County is equivalent to the Queen Sand of New Mexico, and the San Andres formation of Artesia Field of New Mexico is to be correlated with the lower part of the Delaware Sandstone. It is further considered that there is a transition from anhydrite on the eastern margin of the central basin to limestone in the White Horse formation on the western margin, this change being due to the relative heights of the platform during deposition. V. C. I.

1259. Bearing of Cap Rock on Subsidence on Clay Creek Salt Dome, Washington County, Texas, and Chestnut Dome, Natchitoches, Louisiana. (Discussion.) *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1105–1113.

In a contribution to the discussion of cap rock phenomena on salt domes, M. I. Goldman states that the cap rock is indicative of extensive solution of the top of the salt cores. The net movement of these caps is an algebraic result of the upward movement of the salt plug and the downward solution of the salt top, and these factors may explain the complicated evidence of upward and downward movements in the Clay Creek salt dome. The process of solution of the top of the salt core may be traceable to the presence of a more permeable water-bearing horizon in the formations penetrated, but there

is also the possibility of upward diffusion and the attack by water percolating slowly through the overlying beds.

The function of faults in guiding underground waters requires careful consideration, for in the absence of such features it may be questioned how far circulation along permeable beds can be admitted as a possibility.

V. C. I.

1260. Apropos des Méthodes Magnétiques Employées Jusqu'à ce Jour dans la Recherche des Gisements Pétrolifères. A. Belluigi. *Rev. Pétrolifère*, 1931 (443), 1281–1284.

The paper deals with the explanation of some of the anomalies often found in the interpretation of the results of a magnetic survey, where geological data are available for checking.

The author deals with the application of Poisson's principles, where possible, and goes on to evolve formulæ of his own, based on some he has used in gravimetric surveys. In this method he resolves his mass into a series of vertical parallelopipeds, with a horizontal section dx. × dy, and shows that the resultant formulæ can be applied in cases where it is not possible to apply Poisson's. He gives examples of their application in a variety of cases, and opens up a new field of application to this section of geophysics. G. R.

1261. Practical Rules for Determining the Constants of a Magnetometer. C. L. Alexanian. *Ann. des Comb. Liquides*, 1931, **6**, 261–267.

A knowledge of the constants of a magnetometer, when used for geophysical surveying, is of primary importance. Such constants should be determined periodically, and in this article simple rules are given to enable the actual users to determine the constants for themselves.

Methods are given for obtaining the constants of the Lloyd-Schmidt and the Haalck Universal Magnetometers. C. C.

Field Technology.

1262. Killing the Kettleman North Dome Discovery Well. W. A. Sawdon. *Petr. Eng.*, 1931, **2** (13), 65.

Owing to the excessive amount of gas being produced with the oil from several wells in the North Dome of Kettleman Hills it was recently decided to kill or shut in these wells for an indefinite period. The killing of at least two of the wells presented problems necessitating considerable skill and a well-defined plan of procedure.

The equipment for the discovery well, which was the well presenting the greatest difficulty, was specially designed for the work, and the method employed indicates the advancement made in recovering control of deep, high pressure wells.

This discovery well had reached a depth of 7236 ft. when it blew in and sanded up. While clearing out at about 7100 ft. it blew in again with a tremendous amount of wet gas accompanying the oil. About 5300 ft. of 3½ in. drill-pipe with a 5½ in. rock bit were left in the hole and the production from the well was coming around this string.

The oil flowed from the 6⅝ in. casing through two beans into a gas trap, and the production averaged about 3600 brls./day with a flow pressure of 1200 lb./sq. in. Just prior to killing, the production had been cut down to

about 1158 brls./day, but the volume of gas coming with the oil was 51,580,000 cu. ft./day, making a gas-oil ratio of nearly 45,000 cu. ft. of gas/brl. of oil. This gas had an actual gasoline content of 1·28 gals./1000.

The 11 in. casing at this well extended above the derrick floor for 22 ft. with an 11 in. gate valve at the top and a 6 in. valve screwed into a swaged nipple above that. The flow connections could not be disturbed, and so a platform was erected above the 11 in. valve.

A solid concrete foundation was made and the pipe and platform were anchored to both the block and to the derrick corners. The four main vertical members were 10 in. H columns on which were placed 15 in. I beams. On these rested 20 in. I beams which carried the standard Hydril rotary table. Above this was constructed the platform. This platform, designed for a total load of 200,000 lbs., was 30 ft. 9 in. above the derrick floor.

On this platform were placed the lever bank and throttle. Any turning of the table could be done by the catline, but provision was made for rotating if the necessity arose, by putting in an extra bevel gear box. Actually there was no necessity to rotate the table, and the drive shaft to the table was never installed.

Above the 11 in. valve an emergency packer was placed, then a short nipple and then a double or top packer. This top packer was used for running the flush drill-pipe into the hole under pressure. The emergency packer was installed in case it became necessary to change the top packer rubber with the drill-pipe in the hole.

Snubbing-in equipment, consisting of a line-operated pull-down yoke, was rigged above the table and was operated by the sand-line drum. Automatic slips gripped the pipe in either a downward or upward direction, automatically allowing the pipe to be run in and preventing it blowing out and giving a reverse automatic operation when it was being pulled out.

A standard pin on a string of 3½ in. flush-joint drill-pipe was run in and hydraulic pressure applied to the top packers. The 11 in. valve was then opened and the first 60 ft. stand of pipe forced into the hole by means of the hydraulic rotary table. When the weight of the string was sufficient to overcome the pressure in the well this was dispensed with.

On reaching 1700 ft. the pipe encountered the 3½ in. drill-pipe which had been left in the hole. Attempts to take hold were unsuccessful, and the string was removed and an impression block run. As a result a special pin was constructed provided with a guide to ensure its proper contact with the box. The string was run again and connections made with the old string.

After testing the joint, water was pumped through the completed drill-pipe. This water killed the well in 1 hr. 35 mins. and was later replaced with mud fluid. With the well under control the old pipe was easily withdrawn.

In order to ensure that the water would reach bottom in case it would not pass through the old string, a safety joint was inserted and a packing plug was ready for running. The plug would have been run on the end of the flush-joint pipe and expanded above the top of the old drill-pipe. Water would then have been pumped down through it under whatever pressure might have been necessary. L. V. W. C.

1263. Electric Mud Pump Problem Solved. *Petr. World*, 1931, **28** (9), 24.

Recently-completed experiments involving the use of A.C. electric mud pumps with steam characteristics indicate a more general adoption of electric drilling.

In the past A.C. driven mud pumps have had four serious objections, and these have handicapped to a great extent the electrical operation of drilling wells.

These objections are: 1, Lack of flexibility for certain operations; 2, possibility of developing excessive and destructive pump pressures; 3, difficulty in securing satisfactory compound or parallel operation; and 4, tendency to deliver a constant volume of fluid regardless of pressure.

All these objections are directly traceable to the tendency of the electric motor to run at constant speed regardless of pump pressure.

Pump pressure and volume are the main points, but as pump volume is proportional to the motor speed and pump pressure is proportional to the motor torque or twisting effort, the successful operation of the pump depends upon obtaining correct results from the driving motor.

Providing the pump stalls at a definite pump pressure the results would be satisfactory, but this result has not been realised in the past.

These objections have been examined and the control unit has now been changed so that the motor slows down as the pump pressure increases.

Each pump was supplied with an additional permanent resistance in the secondary circuit of the motor, the pump pulley was reduced in size and a motor-driven blower was installed. The blower conducted away the additional heat generated in the motor and compensated for the reduction of the self-cooling action caused by the slowing down of the motor.

It was found that the pump could remain stalled for one or two hours without seriously overheating the motor, and this period was considered satisfactory.

Data showed that the motors had the necessary characteristics for satisfactory parallel and compound operation, and this was verified by actual operation over a wide range of pressures.

The efficiency of the motor was sacrificed somewhat to gain the flexibility and satisfactory service and an overall electrical efficiency of 67% was obtained at a working pressure of 175 lb./sq. in., and a pump speed of 40 strokes/min.

Graphs are given to show at what pressures liners should be changed and whether the pumps should be operated in parallel or in compound to meet any given combination of pressure and volume.

If more volume is required the two pumps may be operated together or the motor sizes increased. In other words, by using sufficient motor capacity with properly proportioned electrical control any conditions of pump pressure and volume may be met. L. V. W. C.

1264. Arc Welding as Applied to Oil Industry. J. C. Lincoln. *Oil & Gas J.*, 1931, **30** (19), 37.

The increasing popularity of electric arc welding in the petroleum industry is a result of the solution of various chemical and metallurgical problems raised by phenomena within the arc itself. In the past electric arc welds were characterised by oxidation, which gave the material lower ductility and tensile strength than ordinary mild steel plate. This was caused by the formation of oxides and nitrides resulting from the action of the atmosphere surrounding the arc on the steel being welded. This has been circumvented by the use of shielded arcs, in which the electrode is heavily coated with a special composition which burns in the arc slower than the electrode itself, forming a sort of crucible which shields the arc for most of its length. The coating probably gives off CO and CO_2, which improve the physical quality of the steel, while the welding speed is considerably improved. The most successful type of weld for oil pipe-lines has been found to be the double bell joint with a backing-up ring, although the plain butt-end type without backing-up ring is frequently used. Arc welding is also extensively used in welding storage tanks and repairing leaks and weak spots in pipe-lines. A description of the manual operations involved in electric arc welding of pipe-lines and repairing of old lines is given. C. L. G.

Crude Oil.

1265. Peruvian Crudes Vary in Properties. G. Egloff, E. F. Nelson and P. Truesdell. *Oil & Gas J.*, 1931, **30** (20), 22.

The production of crude in Peru is recorded from 1896 up to 1930 from official figures. The industry began long before 1896. Almost all the industrially developed fields are situated in N. Peru, and the names of the fields and operating companies are given. In the Zorritos field oil is obtained from depths of 200/1700 ft. which has an average A.P.I. grav. of 38°, is a mixed base crude of agreeable odour and contains 0·04% of sulphur. The yield of products is said to be 13% of gasoline, 40% of kerosine and 40% of residue. The Lobitos and Restin fields produce oils of similar character from an average depth of 3000 ft. Restin crude has an A.P.I. grav. of 34·7° and a sulphur content of 0·05%, Lobitos an A.P.I. grav. of 35·8/37·9° and a sulphur content of 0·06%. The analysis of one Lobitos crude is given and the yield of products said to be: Gasoline of E.P. 392° F., 32·4%; kerosine, 13·1%; gas oil, 14·5%; wax distillate and semi-solid steam refined bottoms (from which wax and lub. oils may be prepared), 38%; coke, 9·4%; loss, 2%. Lagunitas and Negritos oils resemble Lobitos oil, but the La Brea and La Breita fields yield a heavier crude, having an A.P.I. grav. of 18° and a smaller gasoline content. Sulphur contents in all cases are low. Oil has been found in Southern Peru, but production is not of industrial importance. Hydrocarbons isolated by Seyer from Lobitos crude are mentioned. An extract from the official bulletin of the Peruvian Government, giving a list of refineries and their capacities, is quoted. Peruvian crude of A.P.I. grav. 36·4° has been analysed and distilled to produce gasolines of E.P. ranging from 375° F. to 437° F., and the A.S.T.M. distillation of these fractions tabulated. When making gasoline of E.P. 392° F. the yields obtained were: Gasoline, 32·3%; kerosine, 11·0%; gas oil, 16·7%; reduced crude, 39·8%; loss, 1·2%. The properties of the products are tabulated. Topped crude, after removal of the gasoline only, and the reduced crude were submitted to cracking by the non-residuum process under pressures of 150 lb. The pressure distillates produced were distilled to yield gasolines of various E.P.s with the following results:—

	E.P. 392° F.	E.P. 400° F.	E.P. 437° F.
% yield from topped crude	47·9	50·5	62·1
% yield from reduced crude	53·7	55·5	60·5

The distillation ranges of the gasolines and of the pressure distillate bottoms are given. The gasolines were refined with acid and sodium plumbite solution. The antiknock values of the soda-treated straight-run gasoline, and the refined cracked gasolines having in each case an E.P. of 392° F. have been determined in a 8.30 engine. The Octane numbers reported are: Straight-run, 69; cracked from topped crude, 74; cracked from reduced crude, 74.

R. A. E.

1266. Properties of Typical Crude Oils from the East Texas Field. E. L. Garton. U.S. Bur. Mines, Rept. Invest. 3130, August, 1931.

The samples of crude oil under consideration consist of four from the Joiner, Kilgore, and Longview fields of E. Texas, and one from the Van field of E. Central Texas.

All four crudes from E. Texas are of typical "intermediate base" type, produced from an average depth of 3600 ft., the deepest Woodbine Sand production so far met with in Texas or Louisiana. Their general characteristics

are similar. The average sulphur content is 0·28%, total gasoline and naphtha averages 36·0%, kerosine distillate 9·6%, gas oil 15·1%, non-viscous lubricating distillate 9·7%, and medium lubricating distillate 6·2%. The gas oil appears to be a good cracking stock for gasoline production, and the lubricating distillates parallel to those obtained from high-grade refining crude.

The sample from Van field has a higher gravity, and is classed as "paraffin base." Its sulphur content is 0·89%, and its gasoline and naphtha fraction is only two-thirds that of the E. Texan crudes.

Tables showing physical and chemical properties and full analyses of the crudes are presented. C. E. H.

1267. Treating Crude Oil. H. Hoppe. U.S.P. 1,818,480, 11.8.31. Appl., 24.5.29.

Salt water is separated from crude oil by spraying the oil upon a surface heated to a sufficient temperature to explode the globules of oil and water. The mixture of oil and salt water is then delivered into the bottom portion of a body of heated salt water, through which the crude oil will rise to be drawn off from the surface. Gas is sprayed into the oil and water in order to agitate them, and the oil is filtered by means of spaced superposed filters as it passes through the body of salt water. W. H. T.

1268. Treating Crude Petroleum. H. T. Bennett. U.S.P. 1,820,295, 25.8.31. Appl., 22.7.26.

A process is described for treating crude petroleum so as to improve the flowing ability at low temperatures.

Crude petroleum of the type which becomes solid at 60° F. is mixed with 0·1% by weight of a metal soap which is capable of lowering the pour point of the oil and of rendering it capable of flowing at 0° F. W. S. E. C.

1269. Treating Emulsified Oil. F. C. Baker, R. J. Fleckenstein and L. M. Landman, Assrs. to Texas Pacific Coal and Oil Co. U.S.P. 1,821,133, 1.9.31. Appl., 16.11.27.

The patent describes an apparatus for separating water from crude oil. It comprises a flow tank, a line connected to this at a point spaced from the top, the line and tank being arranged to permit the oil to flow through the line at hydrostatic pressure. A heating coil is provided of pyramidal form, formed by a series of horizontal loops of different sizes arranged in tiers. The coil is connected to the line and arranged to permit the oil to flow there through under hydrostatic pressure. Means are provided for applying heated gases directly to the coil, the latter being connected to a separating tank. A conduit is provided for discharging oil from the upper portion of the separating tank and another for discharging water from the lower portion. W. H. T.

See also Abstract No. 1295.

Gas.

1270. Catalytic Gas Reactions. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 353,622, 30.7.31. Appl., 6.6.30.

A process is described for carrying out catalytic gas reactions in which an alkali metal or alkaline earth metal exerts an activating action on the catalyst proper. It comprises bringing the gas to be subjected to the catalytic reaction into contact with the catalyst in the presence of the vapour of at least one of the said metals. W. H. T.

1271. Condensate from Gases. N. V. de Bataafsche Petr. Mij. E.P. 355,413, 27.8.31. Appl., 16.6.30.

The patent refers to the production of a condensate from such gases as natural gas, cracking gas, distillation gas, etc. The process comprises the treatment of the gases by compression in the presence of an added substance. The latter consists exclusively of a component having a boiling point below that of the lowest boiling of the desired components. Also, it contains a considerable proportion of a component which is condensable under the conditions of the condensation. Any undesired lower boiling components are finally removed from the condensate. W. H. T.

Motor Fuels.

1272. Twenty-Third Semi-Annual Motor Gasoline Survey. E. C. Lane, E. L. Garton and A. J. Kraemer. U.S. Bur. Mines Repts. Invest., 3129 and 3142.

The analyses of 312 samples of premium-price and competitive-price gasolines sold in each of 20 cities in the U.S.A. during March, 1931, are presented in tabular form, together with averages for each city. The first report deals with gravity, Reid vapour pressure and distillation data, and the methods employed in taking, handling and analysing samples. The average vapour pressures are higher than those of samples taken in August, 1930, by about 1·9 lb. for competitive-price gasolines and 1·5 lb. for premium-price gasolines, probably as a result of seasonal variation, although part of the difference may be due to losses incurred in handling the August samples. 90·7% of all the samples tested had vapour pressures between the limits of 6·6 and 11·5 lb. Reid. The temperature at which 10% is evaporated on distillation (determined by adding distillation loss to recovery at each temperature) is also shown to vary according to season, the range for March, 1931, being from 88/160° F., as compared with 118/163° F. for August, 1930. Absence of data for samples taken in January, 1930, prevents comparison with those taken in March, 1931, in respect of vapour pressure and the temperatures at which 10% evaporated on distillation. The second report gives the detailed results of other tests and a comparison of the properties of the gasolines with those required by the U.S. Government motor gasoline specification. No sample showed appreciable copper-strip corrosion, only two samples failed to meet the distillation requirements (solely in respect of percentage distilling to 200° C.), 32 samples of competitive-price gasolines failed to meet the sulphur requirement of 0·1% maximum, and 4 samples possessed vapour pressures in excess of the limit of 12 lb. Reid. R. A. E.

1273. Use of Ternary Mixtures as Aviation Fuels. C. Baron. *Ann. des Comb. Liquides*, 1931, **6**, 155–168.

Crystals of ice may be deposited in the carburettors of aero engines flying in low temperatures. The crystals are formed principally round the jets and may lead to serious engine troubles. The effect is due to condensation of atmospheric moisture by absorption of heat by the evaporation of the fuel. It has been found that the addition of alcohol to a benzole-gasoline mixture reduces the tendency to deposit ice crystals to which benzole mixtures are very liable. Tables are given showing the cloud points of various mixtures of these three components, using 99% to 97% alcohol, "motor benzole" and a light aviation spirit.

The cloud point varies with the strength of alcohol employed and with the relative amounts of alcohol, gasoline and benzole. With fixed quantities of 99% alcohol (*e.g.*, 10%, 15%, or 20%) the cloud point becomes lower as the benzol increases from 5 to 30%. In the case of 98% alcohol with 60% of gasoline, the lowest cloud point occurs with 15% to 20% of alcohol, increasing again as the amount of alcohol increases and of benzole decreases.

The use of French-produced alcohol and benzole is recommended in admixture with gasoline, for aviation purposes, the anti-detonating value being increased as well as the tendency to deposit ice crystals being reduced by such additions. E. B. E.

1274. Higher Octane Numbers Bring Premium. R. C. Conine. *Oil & Gas J.*, 1931, **30** (21), 74.

The anti-knock value of the regular gasoline sold at pumps by six of the larger companies operating in Chicago has been increased from octane numbers of 58/60 to octane numbers of 62/67. A more definite scale of price differentiation based on anti-knock value is being advanced in the gasoline tank car markets. The following octane number ranges are at present being used as a basis for this purpose as far as spirits complying with U.S. motor gasoline specifications are concerned: (*a*) Ordinary grade, having an octane number below 57; (*b*) having an octane number of 57/64; (*c*) having an octane number of 65 or over.

Another proposed scheme for price differentiation, based on octane numbers, and considered to be an improvement on the present system, is described and discussed. R. A. E.

1275. Selling Gasoline on Quality Basis. *Commer Stand. Monthly*, 1931, **8**, 50.

Twenty-five States now specify the quality of gasoline which can be sold within their borders. Thirteen of them require the gasoline to meet the Federal specifications. In Colorado gasoline must not be coloured except to identify special qualities, which must be certified by an inspector before a licence is granted. In North Dakota a harmless dye may be used to colour gasolines having an anti-knock value equal to an octane number of 60, but gasolines below this standard must not be so coloured. Gasoline improvers or dopes, except those favourably reported on by national U.S. authorities, may be prohibited if the State authorities consider the claims made are unsubstantiated or the dope detrimental to public health or to motors. The sulphur limit is raised to 0·20% in Montana, and in Illinois gasoline not meeting Federal specifications may be sold if labelled "substandard." Twelve States have specifications not corresponding with present Federal specifications. Some have retained old Federal specifications, others allow an E.P. of 450° F. or have other variations. The names of the States in each category are given. R. A. E.

1276. Vapour Pressure Limit Included in New Aviation Specifications. *Oil & Gas J.*, 1931, **30** (16), 90.

It is proposed to include a vapour pressure limit in the revised specifications for fighting and domestic grades of U.S. Government aviation gasoline. This, together with retention of the acid residue specification, will handicap fuels prepared from natural gasoline. The views of interested parties before the change is made complete are asked for by the chairman of the Federal Specifications Board. The following revisions are proposed for fighting grade: (1) Corrosion test, 100 ml. of gasoline evaporated in a polished copper dish shall cause no grey or black corrosion; (2) on subjection to accelerated

ageing test at 100° C. with oxygen at 100 lb. pressure for 4 hr. and evaporation of 100 ml. of mixture of oxidised sample and gum solvent, the residue shall not exceed 10 mg.; (3) distillation range: at 65° C. at least 10% evaporated, at 100° C. at least 50%, at 135° C. at least 90%, and residue not to exceed 2%; percentage evaporated at any temperature equals distillation loss + amount collected in receiver; (4) vapour pressure—A.S.T.M. method D.323–30T—not to exceed 10 lb./sq. in., or when used for airplanes, etc., not adequately protected from vapour lock, 7 lb./sq. in. Adequate protection requires submerged fuel pump remote from engine, gravity feed maintaining a head of at least 1·5 lb./sq. in., notwithstanding vaporisation of fuel, or other system giving equal protection; (5) freezing point not higher than —60° C.; (6) distillation residue not to show acid reaction; (7) doctor test eliminated. Similar changes which are proposed in the specification for domestic grade are detailed, vapour pressure limits being the same for fighting grade.

R. A. E.

See also Abstract No. 1296.

Lubricants and Wax.

1277. Lubricating Oils from Paraffin-Free and Paraffinic Crude Oils. H. Ordelt. *Petr. Zeit.*, 1931, **27**, *Motorenbetrieb.*, **4** (7), 3–9.

Temperature-viscosity curves have been plotted from results obtained for different types of paraffins and ceresines, and these are found to be independent of the fractionation of the paraffin, but dependent on their setting point.

The oil with the greatest Tansz exponent as denoted by x in the equation

$$\eta^x \text{ oil diluted} = \eta \text{ oil diluted}$$

has the flattest viscosity curve. American paraffinic oils are members of this class and paraffin-free oils have the least exponent. The Tansz exponent can be increased to an optimum by adding paraffin to the low set-point paraffin-free oils or oils poor in paraffin whose curves are flat.

The type of refining is found to have no influence on the temperature-viscosity curve or on the Tansz exponent. W. S. E. C.

1278. Diesel Engine Lubrication. C. S. Darling. *Mar. Eng.*, 1931, **54**, 368–369.

The types of oil required are crankshaft, camshaft, cylinder and compressor oils, and their functions vary with the engine. In a normal 4-str. cycle engine the oil fed to the bearings serves as a lubricant only, but the oil supplied to the bearings of a 2-str. cycle engine serves also as a gas-seal, preventing escape of air from the crankcase.

With vertical oil engines, experience indicates the advisability of using individual oil feeds to the pistons, but with horizontal engines sufficient lubrication is obtained with only one oil feed, the lubricating oil pumps being preferably of the multi-ram positive displacement type. Air injection engines require a separate oiling system for the compressors, and the compressor oil should have a good margin of safety, particularly as regards flash point. Where compressors are such that the major portion of the work comes from the high pressure stages, the ratio of compression is considerably increased, and with it the final temperature of compression. An increased margin of flash point over normal working temperatures is essential, therefore, unless extra cooling

of the cylinder jackets is provided. Double-acting engines involve the oil for cylinder lubrication in three directions: the upper and lower cylinder piston rings and the piston-rod gland, the oil in the latter serving also as a seal and cooler for the gland. It appears necessary for the oil from the gland to be kept separate from the other oil returns.

The oil supplied to the piston is subject to change of properties, due to the contact with combustion gases. The rise of the temperature during compression would cause destruction of the oil film if it were not wholly covered by the piston before it has risen to, or exceeded, the flash point. Oil carbonisation does not interfere with its lubrication qualities because the oil has done its work before the destructive action of the gases begins. It is indicated, also, that a more viscous oil is needed with double-acting engines because of the greater frequency of the period of maximum temperature. A discussion follows on the centrifugal system of oil reclamation at temperatures of 100–150° F., together with an indication of desirable features of oil coolers. With a single fleet of ships returning to one port, suggestions are made about a central depot for oil storage and filling and also for oil reclamation. (The author appears to have confused "flash point" with "spontaneous ignition temperature" in his discussion of compressor oils.—Abstractor.)

C. I. K.

1279. Factors Controlling the Effective Lubrication of Diesel Engine Power Cylinders. A. M. Tode. *Petr. Eng.*, 1931, **2** (13), 94.

The selection of the correct lubricant for a Diesel engine is an extremely important factor in obtaining efficient operation. The fundamental requirements of the engine itself are: cleanliness of design, simplicity and ready access to all parts, and that the lubrication system should be thorough, but as little complicated as possible. The modern trend is to lubricate as far as possible by mechanical means only. The necessary characteristics of lubricants to be used for cylinders, air compressors or bearings are: (1) Careful refinement and freedom from comparatively volatile fractions; (2) low emulsification tendency; (3) suitability for the type of engine and lubricating system installed; (4) a viscosity sufficiently high to maintain an adequate film between wearing surfaces at operating temperatures, but not so high as to create abnormal internal friction; (5) sufficient adhesiveness to resist squeezing out from wearing surfaces at normal operating pressures; (6) a pour test low enough to avoid necessity for heating storage tanks; (7) a capacity to spread readily over wearing surfaces, particularly in the case of cylinder wall lubrication, to prevent inadequate piston sealing; (8) low carbon residue content.

The lubricating demands met with in the Diesel engine are: (1) High rubbing velocity with moderate pressure; (2) low rubbing velocity with high pressure; (3) low pressures with intense heat; (4) small, lightly loaded parts requiring a complexity of feeds. Although the demands are diverse in character, recent tests have shown that in many cases one grade of oil can be chosen to meet all requirements, especially for engines of small size. Oils should be selected on a basis of viscosity and freedom from impurities. Various lubrication systems are described and illustrated. Successful Diesel engine cylinder lubrication is dependent on four factors: (1) Use of properly refined oil; (2) application of sufficient, though never excessive amounts; (3) delivery through oil ways so located that the piston and rings receive the maximum of this charge; (4) application at exactly the right time. Oil should be delivered regularly to cylinders and pistons by some form of force feed lubrication which will insure a charge of sufficient volume to cover the entire cylinder wall under the swabbing action of the piston. The differences

between the lubricating requirements of 2-str. and 4-str. Diesel engines are enumerated. Temperatures reached in the cylinders and by the cylinder walls are given, and the effect of the exposure of the lubricant to these temperatures discussed. R. A. E.

1280. Dewaxing Lubricating Oils by Distillation. E. Pyhälä. *Erdöl und Teer.*, 1931, 7, 446.

For the treatment of paraffin-naphthene base oils or those containing low m.-pt. paraffin, the author recommends distillation as against the generally more costly and complicated method of cold filtration. As the former method seems little known, he gives a brief description of the process, using superheated steam at ordinary pressures. A specially-constructed "condensing dephlegmator" is employed. From the cooler are drawn off dewaxed oils, the paraffin and light oils remain as an easily pressable mass, while in the still is left a highly viscous oil of good cold test. The distillates from the cooler may be further fractionated. They are quite bright and easily refined. The separation thus obtained is stated to be very good. It is emphasised that the dephlegmator employed is quite different from those ordinarily used in the petroleum industry, but no details are given of its construction. E. B. E.

1281. Domestic [U.S.A.] Consumption of Lubricating Oil. D. J. Gill. *Oil & Gas J.*, 1931, **30** (21), 52.

The refinery value of lubricating oils has approximated 225 mil. dollars annually in recent years. This value greatly exceeds that of many products for which basic economic data are published by the Federal Government. Although various statistics are published monthly in regard to lubricating oils as a whole, it is thought that, in view of the widely differing nature of such oils, sub-division into groups of oils of similar characteristics is necessary, and would provide data of immense value in following changes in demand, in value, etc. This theory is developed and illustrated by examples of factors which have influenced price structures, consumption and manufacture. Various graphical illustrations from data now available are provided and explained. They show, among other things, that the variations from the trend of indices of business activity, and the variations from trend of shipments of industrial oil made by a representative oil company, provide curves of similar nature over the period 1923/1931. Similar curves, including estimated variations from trend in the domestic consumption of motor oils and industrial oils, are provided. The basis used for the estimated annual changes in consumption of industrial and automotive lubricating oils is explained and elaborated.

Since lubricating oil enters intimately into every major and most secondary economic operations of our time, it may be concluded tentatively, from the limited data available, that the value of industrial lubricants currently consumed, related to a carefully determined trend, probably presents the best available single indicator of general business conditions. R. A. E.

1282. Production of High Quality Lubricating Oils. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 354, 441, 13.8.31. Appl., 28.5.30.

Liquid products obtained by cracking paraffin waxes and which have an iodine value of above 150 are condensed in the presence of catalysts to give lubricating oils. Anhydrous halides are quoted as examples of catalysts. The conditions of cracking are determined by the nature of the initial material, but pressures of the order of 30 atm. and a temperature of about 500° C. is suggested. During condensation a rapid rise in temperature takes place, and this should be regulated. It is often desirable to add cyclic hydrocarbons to the cracked products prior to the condensation. W. D. S.

1283. Refining Lubricating Oils from Heavy Hydrocarbon Oils by Treatment with Hydrogen at High Temperatures and Pressures. Standard Oil Dev. Co. E.P. 354,478, 13.8.31. Appl., 18.6.30.

High boiling unrefined hydrocarbon oils are heated to a temperature between the approximate limits of 700° F. and 850° F., in the presence of a gas rich in free hydrogen under a pressure of about 200 atm. Catalysts may be employed. The oil is thus hydrogenated and conditions are so adjusted that less than 20% of the volume of the feed oil is converted into a liquid product boiling below about 400° F. Mixtures of catalyst and reaction mixture are continuously withdrawn and distilled to give several cuts, one of which is a high-grade lubricating oil. The fluid residuum containing the catalyst is returned to the reaction zone. W. D. S.

1284. Production of Wax Compositions. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 354,782, 6.8.31. Appl., 6.5.30.

The wax compositions comprise one or more waxes, a substantial quantity of hydroxy-alkyl amines and/or derivatives thereof and organic solvents, and, if desired, diluents which do not dissolve the waxy materials. At least one of the waxes is a crude, bleached or purified Montan wax or a derivative thereof. W. H. T.

1285. Separating Wax from Mineral Oils. H. T. Bennett, Assr. to Atlantic Refining Co. U.S.P. 1,820,645, 25.8.31. Appl., 9.1.30.

This patent describes a process for separating from mineral oils various waxy substances including the naturally-occurring paraffin wax, amorphous wax, petroleum or hydrocarbons which normally solidify or become semi-solid below a temperature of about 190° F.

The mineral oil is dissolved in a mixture of petroleum naphtha and a crystalline organic material containing diphenyl and the resultant solution is chilled to a temperature low enough to precipitate some of the waxy substances and diphenyl. W. S. E. C.

Fluxes and Asphalts.

1286. Road Emulsions. C. L. McKesson. *Erdöl und Teer*, 1931, **7**, 448–449; *Canad. Engineer*, 1931, **18**, 15–17.

The author divides emulsions into two classes—"mixing-emulsions" and "penetration-emulsions." The former must be capable of dilution, must easily wet stone chips and must not break quickly, in order that proper distribution of the asphalt may be obtained. They usually contain 65–70% asphalt. The viscosity is of little importance, unless the emulsion is to be laid with a spraying machine when the asphalt content is only 55–60%. "Penetration-emulsions" are used in special cases. After laying they must be left until the asphalt particles have coagulated. In wet weather there is a tendency for the emulsion to be washed away. The required viscosity depends on the amount of penetration desired. 50–55% of asphalt usually gives good results for deep penetration, and 55–60% where thinner layers are to be treated, a higher viscosity being here permissible. A description of some of the tests employed is then given. For "penetration-emulsions" a "washing-test" is employed, consisting of coating 2 kilos of $\frac{1}{4}$–$\frac{1}{2}$ in. gravel with the emulsion, washing with water until the water comes through clear,

drying and determining the amount of asphalt left on the gravel. Quick-breaking emulsions leave 80–100%, and "mixing-emulsions" 2–40% of asphalt. Results of experiments on emulsions with varying amounts of emulsifier are tabulated. Meyer's de-emulsification test, as modified by the American Bitumuls Co., is also described. 100 grms. of emulsion are treated in a weighed beaker with 35 c.c. 0·02N calcium chloride during two minutes, the emulsion then being poured through a weighed No. 14 sieve. After exhaustive washing the beaker and sieve are dried and weighed; and the percentage of asphalt left is termed the emulsification-index. Quick-breaking emulsions give indices of 80–100%. For "mixing-emulsions" 50 c.c. 0·1N calcium chloride is used. Good agreement is obtained between this test and the wash-test, as is shown by curves and tables. A "sedimentation-test" consists of leaving 500 c.c. of emulsion contained in a glass cylinder for 10 days and determining the difference in asphalt content of the top and bottom 50 c.c. This should not exceed 6%. The Saybolt-Furol viscometer is recommended for determining the viscosity of emulsions. Other tests employed are as given by the A.S.T.M. E. B. E.

1287. Separating Bitumen from Bituminous Sands. C. T. Langford and A. J. Teplitz, Assrs. to Continental Oil Co. U.S.P. 1,820,917, 1.9.31. Appl., 29.4.27.

A solution of sodium hydroxide is added to a bituminous sand and the mixture subjected to an abrasive grinding action at a temperature of approximately 70° C. The mixture is then drawn off and water added to effect the final separation of the bitumen from the sand. W. H. T.

1288. Treating Aqueous Emulsions of Asphalt. Metallgesellschaft A.-G. E.P. 353,702, 30.7.31. Appl., 28.7.30.

The patent describes a process for breaking aqueous emulsions of asphalt, etc., which have been stabilised by the addition of soluble phosphates (*e.g.*, tertiary sodium phosphate). After application during road making, the emulsions are subjected to the action of solutions of salts, the bases of which form insoluble or sparingly soluble compounds with the phosphoric acid ion of the emulsifying agent. W. H. T.

1289. Treating Asphalts. H. D. Elkington, Assr. to N. V. de Bataafsche Petr. Mij. E.P. 355,430, 27.8.31. Appl., 30.6.30.

This invention concerns a treatment of asphalts which are sticky in character, whereby such stickiness, which is disadvantageous when employed in asphalt enamels, etc., can be removed. The treatment consists in adding to the molten asphalt siccative substances (3–10%), comprising resinates, sulphonates, naphthenates, linoleates or the like salts of the heavy metals, *e.g.*, manganese, cobalt, lead-manganese, lead, copper, zinc, uranium, or mixtures thereof, with salts of other metals, such as alkali or alkaline earth metals. W. H. T.

1290. Bituminous Compositions. Amber Size and Chemical Co., Ltd. E.P. 356,003, 3.9.31. Appl., 13.10.30.

A process is described for the production of bituminous substances, liquid at ordinary temperature, utilisable in any weather and of greatly increased wetting capacity for road construction purposes. To bituminous substances other than tar, there are added materials which contain oxy compounds of the aromatic series, and hydrocarbons as well as nitrogen bases such as aromatic amines, and also azocyclic compounds, *e.g.*, pyridine. The materials are oily products obtained in the destructive distillation of recent or fossilised animal substances, such as Dippel's oil, bone oil, crude shale-oil or shale tar. W. H. T.

Special Products.

1291. Useful Conversion of Refinery By-Products. L. A. Mekler. *Chem. and Met. Eng.*, 1931, **38**, 538.

In many refineries petroleum coke is burnt not only with a view to eliminating storage difficulties or fire hazards by burning in the open, but also to recover efficiently the heat available from its combustion. Outside fuel users are also using petroleum coke for power generation, metallurgical purposes, etc. One company has built a power plant in the vicinity of a number of refineries producing petroleum coke, using this as the only fuel. Another power company accepts all the carbonaceous waste products of the refinery as part payment for the power supplied. Petroleum coke is an ideal fuel for powdered fuel firing, owing to the absence of trouble caused by ash, slag and corrosion of brick setting. Before burning, fresh coke is weathered for 24 hours to remove moisture, crushed and passed over a magnetic separator, while the large lumps of hard pitchy coke are also removed. An efficiency of 80% for boilers burning petroleum coke can be obtained.

Refinery acid sludge can be treated to remove oil and weak acid, the latter being either concentrated or sold as etching and cleaning acid for metals. Sulphonic and other organic acids can also be recovered from the acid sludge. In many refineries acid sludge is successfully burnt in outside rotary burners. For preference the acid sludge should be neutralised with alkali sludge. Usually sludges have a higher flashpoint and burn more slowly than standard liquid fuels so that high combustion temperatures must be maintained. To prevent coking the burners must be protected from the radiant heat of the combustion chamber.

Residuums containing as high as 40% B.S. and W. can be successfully burnt without admixture.

Refinery gases present no important difficulties in burning, particularly if they are free from light liquid hydrocarbons. Treatment of refinery gas is carried out for the removal of valuable liquid hydrocarbons for incorporation into motor fuel. The dry gases are often fractionated to separate and liquefy propane and butane, which are extensively used as industrial and domestic fuel. Butane is successfully used for welding, and is in some cases reported to give better results than oxy-acetylene.

Cracking or reforming of gases to yield benzol, butyl alcohol, methanol, etc., particularly those suitable as solvent for lacquer and other cellulose products is now in the commercial stage. The halide derivatives of the light hydrocarbons obtained from refinery gases form a series of valuable products important as resins and impregnating materials. C. L. G.

Refining and Refinery Plant.

1292. Petroleum Refinery Effluent Treatment. J. S. Hess. *Chem. and Met. Eng.*, 1931, **38**, 537.

The disposal and purification of liquid wastes presents a serious problem to many refineries. The usual method of preventing the escape of oil with the usual condensing, wash or settled water, is by means of a comprehensive drainage system and one or more gravity type oil separators of sufficient capacity to retain the oil and sediment. The drainage system usually consists of two separate systems of sewers, one for sanitary waste and one much larger

system for oil and water waste. The separator usually used is of the gravity type, consisting of a rectangular box divided into compartments by baffles extending from above the surface of the water to a point about half-way from the water surface to the floor. The oil which rises to the surface is retained by the baffles and skimmed off and transferred to tanks for further use. It is necessary that emulsions should be broken before passing through the separator. Water containing chemicals used in refining is generally treated to reclaim the chemicals, the residue left being rendered harmless by dilution.

Reference is made to the information given in Section 1 of the Manual on the Disposal of Refinery Wastes prepared by the Committee on Disposal of Refinery Wastes of the Division of Refining, A.P.I. C. L. G.

1293. Utilization of Refinery Gases. W. T. Ziegenhain. *Oil and Gas J.*, 1931, **30** (10), 26.

At the meeting of the A.C.S., held in Buffalo in September, a symposium was presented on the utilization of gaseous hydrocarbons. This problem is of considerable importance at the moment, owing to the increasing production of gas resulting from more intensive cracking to produce gasoline of highest octane number, and also owing to the search for sources of revenue from by-products. A paper presented by F. E. Frey and H. G. Hepp describes laboratory and commercial scale results in the pyrolysis of simple paraffins with the object of producing benzol. Owing to the high temperature necessary for its decomposition, methane is not a likely source of benzol in spite of its abundance. Vapours from fractionators composed chiefly of ethane, ethylene, propane and butane are a more likely source of motor fuel. Cracking tests were carried out in a silica tube mounted in an electrically heated furnace, the cracked gases issuing from the tube passing through an electrical precipitator to remove suspended tar and through a condenser cooled with solid CO_2 in acetone to a receiver. The tar collected was re-distilled under vacuum to remove volatile liquid hydrocarbons. Results of actual tests on a gas of composition methane 18.6%, propane 44.7%, n. Butane 32.7% and Isobutane 4.0% when cracked at 850° C. are given. It is shown that the yield of volatile oils varies from 3.0 to 14.2% with varying time of contact, the tar increasing from 0.2 to 14.7%. The primary decomposition of the propane and butanes into simpler paraffins and olefines was nearly complete in 0.0025 minutes, during which oil formation was negligible. The oil forming stages of the reaction consumes a much larger time. During this stage methane formation increased to a high final value, hydrogen increased somewhat, and with the exception of ethylene and ethane the other gaseous hydrocarbons were quickly destroyed. Details of the probable courses of the reactions in connection with the decomposition of the various hydrocarbons are given. It is calculated that 650 calories/gram are required to heat the uncracked gas to the reaction temperature, after which rapid primary decomposition took place with absorption of heat followed by the slower oil forming reactions with evolution of heat.

Other papers presented at the symposium describe the utilisation of natural gas for the production of carbon black by the Thermatonic process. This process gives a yield of resultant gas containing 85.4% hydrogen, and is of particular interest to refineries considering hydrogenation plants.

Reference is also made to the Cyclic Process for the catalytic production of hydrogen from methane. C. L. G.

1294. CONTINUOUS CENTRIFUGAL. E. M. James. *Mech. Eng.*, 1931, **53**, 743–749.

The centrifugal force developed, e.g., in a rotor or bowl revolving about a vertical axis, is given by $F = K N^2 r$

where F = number of times the force of gravity developed.

r = radius.

N = r.p.m.

K = constant = 0·0000284 when r is in inches.

The commercial super centrifuge operates at about 15,000 r.p.m. to deliver a separating force of 13,200 times gravity, and has separation times of from about 7 minutes to 3½ seconds.

The three principal factors involved are firstly the effective mass of the particles of the dispersed phase. Effective mass is the product of actual mass times difference in specific gravity between continuous and disperse phase. Secondly, viscosity of the medium through which particles of disperse phase must move, and thirdly, the time in which a unit mass of liquid is subjected to centrifugal force.

The first and second factors can be controlled somewhat by dilution or by raising the temperature. The super centrifuge has a wide field of application in the removal of dirt and carbon from lubricating and fuel oils. Lubricants from stationary engines are continuously repurified and returned to service, whilst with fuel oils, clogged burners are avoided and low grade oils can be cleaned up.

The dehydration of crude petroleum, containing emulsified salt water, can be effected rapidly and with small evaporation losses, transformer oil can be purified and switch oils reconditioned for use.

The ability of the super centrifuge to handle a three phase system consisting of two liquids and a solid, where the specific gravity of the latter is lower that that of the heavier liquid, is utilised in dewaxing cylinder stocks.

The chilled suspension of wax in cylinder stock-naphtha solution is treated with a carrier solution heavier than the wax and immiscible with the oil-naphtha solution and a special design of centrifugal rotor is used. The wax can be discharged continuously to produce a bright stock of cold test 15° F. (approx.). Long residuum and various heavy distillates can also be handled and more than 10,000 barrels of oil are now being dewaxed daily by the centrifugal in the U.S.A. W. E. J. B.

1295. REFINING OF CRUDE OILS IN GERMANY. E. F. Nelson and G. Egloff. *Oil and Gas J.*, 1931, **30** (16), 30.

Crude oil from the Nienhagen Field, Hanover, has the following properties: A.P.I. grav., 26·6°; sulphur, 0·5%; coke content, 4·6% by wt.; I.B.P., 230° F.; 11% over at 410° F.; E.P., 718° F. By subjecting the oil to cracking in an experimental plant at a reaction chamber pressure of 250 lb./sq. in. and transfer temperatures of 933° F. and 949° F. respectively, the products obtained were:—Gasoline, 50·7 and 57·4%; flashed residue, 43·8 and 34·1%; gas and loss, 5·5 and 8·5%. The gasolines were similar in boiling range:—I.B.P., 128° F.; 31% over at 212° F.; E.P., 380° F., and the anti-knock values when tested in an S.30 engine were equivalent to octane numbers of 73 and 75 respectively. The viscosities of the flashed residues were 108 and 485 secs. Furol at 122° F. Other properties of the residues, and the yields and properties of asphalts of penetration 30/40 and 90/100 prepared from the thicker residue are given. A commercial cracking test has been carried out at the Belgian Cracking Co.'s plant on the same crude,

but using the non-residuum method. The operating conditions were :—Pressure, 225 lb./sq. in. Temperature, 900° F., and the products obtained were :—Pressure distillate, 76% ; coke, 49 lb./brl. Calculated on the original crude, the gasoline which could be obtained from the pressure distillate varied from 55% of E.P. 392° F. to 62·5% of E.P. 437° F.

Crude oil from the Edessa Field, Thuringia, has the following properties :—A.P.I. grav., 34·5° ; sulphur, 0·3% ; visc. Furol at 77° F., 12 sec. ; coke content, 3% by wt. ; I.B.P., 140° F. ; 27% over at 410° F. ; E.P., 740° F. This crude yields 18·7% of str. run gasoline having an E.P. of 350° F. and octane number 49, or 21·6% having an E.P. of 370° F. and octane number 46. The topped crude, after removal of gasoline having an E.P. of 370° F., was cracked at a reaction chamber pressure of 350 lb./sq. in., and at a temperature of 950° F. The products obtained were :—68·8% of gasoline (A.P.I. grav. 62·6°, 20% over at 168° F., E.P. 369° F., octane number 73) ; 20·7% flashed residue (visc. Furol at 212° F. = 61 sec.) ; and 10·5% gas and loss. R. A. E.

1296. Barnsdall Refinery has Combination Recovery and Gasoline Stabilizer. L. S. Gregory. *Oil and Gas J.*, 1931, **30** (19), 28.

The Barnsdall Refineries recently commenced operating a combined recovery gasoline and pressure gasoline stabilizer at their Oklahoma refinery in order to control the vapour pressure of the gasoline tendered to pipe-line systems. The plant can handle 5000 gal. of recovery and 40,000 gal. of pressure gasolines daily. Pressure gasoline is introduced into a surge tank under its own pressure of approx. 20 lb./sq. in., and from there a centrifugal pump delivers it to a feed surge tank under a pressure of 50 lb./sq. in., which is equivalent to the pressure of the gasoline from the recovery plant. A constant level device regulates the flow of pressure gasoline to the feed surge tank, from which the column feed pump takes suction and discharges through heat exchangers into the side of the stabilizer. Overhead vapours from the stabilizer are partially condensed in a reflux condenser and passed to a reflux accumulator, where condensate is separated from stripped gas and pumped to top of the stabilizer as reflux. Stripped gas leaves through a back pressure valve which holds a constant pressure on the system. At the base of the column in a steam-heated reboiler held at constant temperature by a regulator. Stabilized gasoline of the required vapour pressure is removed from the reboiler, passed through the heat exchangers, and thence through coolers to storage. The construction and control of the plant are described and a flow sheet provided. The required vapour pressure of the stabilized product at any time can be ascertained from a knowledge of the vapour pressures, qualities and quantities of the other gasolines available. R. A. E.

1297. Tidal Refining Co. Making High Quality Coke by Knowles Process. W. T. Ziegenhain. *Oil & Gas J.*, 1931, **30** (17), 16.

Petroleum coke of unusually high fixed carbon content is being produced by the Knowles coking process at the Drumright, Okla., plant of the Tidal Refining Co. The process is used to reduce a mixture of topped crude and residue from tube and tank cracking plants, taking off a distillate which passes through a fractionation column giving gasoline of high anti-knock value and a gas oil suitable as intake for the cracking plants.

The particular feature of the Knowles process is the coking ovens. These measure 10 ft. by 30 ft. in area and the floor and sides are lined with tight fitting special alumina clay tiles. The combustion compartment is below the oven, heat being applied by ten gas burners through ten 8 in. by 6 in. tunnels.

The coking process is carried out continuously in the battery of six ovens, the cycle lasting 8 hrs., each oven being 1 hr. 20 min. behind the oven ahead. The intake material from the topping and cracking plants is first passed through a single pass pipe still where its temperature is raised to 800° F., and then enters the coke oven by two ¾-in. lines extending just through the refractory lining and spaced so as to give an even flow of oil over the whole floor of the oven. After striking the hearth, which is maintained at a temperature of about 2,400° F., the oil quickly vaporises, the temperature of the vapours being about 1,300° F. The temperature of the oven gradually decreases as coke is built up on the floor. After about 5 hrs. and 20 min. it reaches a height of 6–8 in. at which point the oil flow is stopped and the drying process commenced. This takes 2 hr., the remaining 40 min. of the cycle being occupied by removing the coke.

After completion of the drying process the doors of the oven are unsealed and raised. The coke immediately ignites and is allowed to burn for a few minutes to remove fine carbon dust, etc. A ram then pushes the glowing masses into a trolley which is run to the quenching station where it is cooled by water sprays and dumped for grading.

A typical run on a mixture of 91·81% topped crude and 8·19% cracked tar gave the following yields: Gasoline (44·0 A.P.I.), 7·93%; Gas oil (20·0° A.P.I.), 55·02%; Gas, 19·62%; Coke, 16·72%; Loss, ·70%.

The coke produced has an apparent specific gravity of 1·12 and true specific gravity of 1·8, moisture 0·6%, fixed carbon 94·8%, ash 1·8% and volatile matter 2·8%. The gasoline distillate has a high anti-knock value, but a yellow colour and high end point. It is therefore treated and rerun. The boiling range is 160/490° F. while the gum content is 200/300 (100 c.c. in copper dish). The gas oil has a gravity of 19·7°, 5% distilling to 700° F. and B. S. & W. 3.3%. Diagrams and a flow sheet of the plant are given. C. L. G.

1298. Two Oklahoma Refineries Install Waste Water Recovery Plants. C. O. Willson. *Oil & Gas J.*, 1931, **30** (18), 16.

In areas where water costs are high or stream pollution is prohibited, the installation of waste water recovery plants is practical. In one Oklahoma refinery working up 4000 brs. of crude daily, the waste water (consisting of water washing of light oils after chemical treatment, tank drainings and steam condensate) amounts to 75,000 gals. daily. The units employed may be of the steel settling tank and pressure filter type or concrete settling basin and gravity filter. In both cases the refinery waste water is collected in a sump where the oil is skimmed off. The skimmed water is delivered to the treating plant where sufficient lime is added to establish the desired alkalinity, followed by alum which yields a heavy floc of aluminium hydrate. The heavy precipitate thus formed adsorbs and absorbs the soluble and suspended oil and other organic matter and is deposited as a sludge which is subsequently removed. The settled water then flows to a sand filter which removes any suspended solids. Owing to the presence of dissolved mineral matter, it is not usually suitable for boiler use without further treatment and owing to its taste and odour cannot be disposed of into a public stream, without purification, e.g., through activated carbon. C. L. G.

1299. Treatment of Hydrocarbon Oils with Liquid Sulphur Dioxide. Edeleanu G.m.b.H. E.P., 354,582, 13.8.31. Appl., 2.9.30.

An improvement in the Edeleanu process is described in which the hydrocarbons to be mixed, as well as the mixing itself, are effected in one and the same centrifugal pump, instead of utilising separate pieces of apparatus for the feeding and the mixing. W. S. E. C.

1300. Refining Crude Petrol. I.C.I., Ltd., W. R. Madel and E. W. Fawcett. E.P., 355,212, 18.8.31. Appl., 18.2.30.

Crude petrol (e.g., that obtained by low temperature carbonisation) according to the invention, may be freed from phenols by cooling to a temperature below —40° C. and removing the material which is precipitated from solution. The cooling process may be carried out on the fraction boiling between about 170° and 200° C., and in the presence of solid adsorbents. W. H. T.

1301. Treating Petroleum Hydrocarbons. R. Cross. Assr. to Cross Development Corpn. U.S.P. 1,817,969, 11.8.31. Appl., 28.8.24.

Hydrocarbon oil is contacted with sulphuric acid and then, without water washing, with inorganic adsorptive material mixed with a metallic compound, the latter having an affinity for sulphur compounds. W. H. T.

1302. Reaction Between an Aqueous Reagent and Impurities in Oil. A. E. Flowers, Assr. to De Laval Separator Co. U.S.P. 1,818,702, 11.8.31. Appl., 9.6.27.

The aqueous reagent and the oil flow respectively into chambers on opposite sides of a porous medium, and a circulation of the oil is maintained into and out of its chamber along the porous medium in a stream which is narrow relative to the length and area of the surface of the stream in contact with the porous medium. Outflow of the aqueous reagent from its chamber is prevented except through the porous medium, sufficient pressure being applied to maintain a high rate of flow. The aqueous reagent thus penetrates the oil of the flowing stream over an extended area of oil in a disseminated state without producing substantial agitation. W. H. T.

1303. Treating Liquid Hydrocarbons. G. H. van Senden, Assr. to Shell Development Co. U.S.P. 1,819,348, 18.8.31. Appl., 27.3.28.

Petroleum distillates containing decomposition products are refined by treating in three stages; in the first counter-currently with acid sludge produced in the second step, said sludge being less than 40° Bé in gravity; the second step involves counter-current agitation of the oil refined by the first step with sludge produced from the third step, this sludge being less than 55° Bé in gravity; and finally the third step consists in counter-current treatment of the oil from step two with fresh acid of from 63–68° Bé, and so limited in amount as to produce a sludge of gravity not over 60° Bé. S. F. B.

1304. Removal of Gas and Low-Boiling Fractions from Petroleum Products. W. G. Whitman and C. C. Miller, Assr. to Standard Oil Co. (Ind.). U.S.P. 1,819,760, 18.8.31. Appl., 9.3.27.

A method of utilising pressure distillate from cracking operations is described. The distillate is cooled by reducing the pressure and thereby liberating gas. The gas is caused to expand in the presence of the distillate, so further reducing the temperature. This cooled distillate is then introduced into the upper part of a fractionating column into an intermediate portion of which the distillate with its contained gas is also introduced. Heat is applied at a lower part of the column. W. D. S.

1305. Heating Decomposable Materials. J. R. Schonberg, Assr. to Standard Oil Dev. Co. U.S.P. 1,821,326, 1.9.31. Appl., 11.6.27.

The patent describes a method for economically heating fluid sensitive to excessive temperatures. It consists in burning fuel with relatively small excess of air in a radiation zone. A layer of semi-opaque gas is interposed

between the flames and heating surfaces containing the fluid to be heated. The gases from the radiation zone are withdrawn, mixed and passed to a second heating zone, the heat therein being absorbed. The semi-opaque gas is at least 1 ft. in thickness, and contains substantially equal parts of CO_2 and water vapour. W. H. T.

1306. Distilling Hydrocarbon Oils and Apparatus Therefor. W. H. Bahlke, Assr. to Standard Oil Co. (Ind.). U.S.P. 1,822,607, 8.9.31. Appl., 4.4.27.

A pipe still is described in which the treated oil is passed in a confined stream through a heated zone whereby the oil receives the entire heating for the process. The vapours and unvapourised oil pass into a fractionating column comprising an equilibrating section and a subjacent stripping section. Means are provided for variation in the method of exchange between these two sections. Diagrams are attached. H. C. R.

1307. Refining Hydrocarbons. J. G. Stafford, Assr. to Sinclair Refining Co. U.S.P. 1,823,175, 15.9.31. Appl., 30.3.28.

The patent describes an improvement in the refining of cracked hydrocarbon distillates which comprises their admixture with a stable liquid hydrocarbon having a higher boiling range and being capable of acting as a solvent for the high boiling reaction products formed in the refining operation. The mixture is subjected in a liquid state to an elevated temperature (375°–500° F.) lower than that at which substantial cracking will occur, and simultaneously to an elevated pressure in the presence of a finely divided solid adsorbent material—e.g., fullers earth. W. H. T.

See also Abstract No. 1291.

Cracking.

1308. Conversion of Petroleum Oils. Petroleum Conversion Corpn. E.P. 354,496, 13.8.31. Appl., 25.6.30. Con. (U.S.A.), 12.11.29.

The process consists in heating up a carrier gas to at least conversion temperature and vaporising a petroleum oil without cracking, then mixing the carrier gas and the vapours in a reaction chamber. The dry oil vapour immediately before mixing is superheated by passing through a superheating zone so rapidly that practically no cracking occurs. The superheated vapour may attain a temperature of 900° F. whilst the cracking zone temperature varies from 950° to 1000° F.

An apparatus is specified for carrying out the operation. R. P.

1309. Apparatus for Decomposition of Petroleum Hydrocarbons. J. C. Black, Assr. to Gasoline Products Co. U.S.P., 1,819,729., 18.8.31. Appl., 29.8.25.

An apparatus for cracking oil is described. The essential features are a supply tank connected to a cracking coil in a furnace, an evaporator and a fractionating tower. Between the supply tank and the coil is interposed a dehydrator and heat exchangers are positioned between the supply tank and the dehydrator. The dehydrator may be by-passed. A transfer line is provided for passing oil from the coil through the secondary heat exchanger

to the evaporator in heat exchange relationship with the oil passing to the coil. The unvaporised residue may be removed from the evaporator and passed through the primary heat exchanger in heat exchange with the oil passing to the coil. The condensate from the fractionating tower may be led back to the supply tank. W. D. S.

1310. Gasoline from High-Boiling Hydrocarbon Oils. J. C. Black and M. L. Chappell. U.S.P., 1,820,371., 25.8.31. Appl., 31.10.27.

The invention relates to the manufacture of gasoline by the cracking of high boiling petroleum oil mixed with hydrogenated oil obtained by hydrogenation of the products of the cracking reaction. The hydrogen required is obtained by decomposition of the gaseous products. Conditions of cracking are 850° F. at 500 lbs., and those of hydrogenation 700–750° F. at 1000 lbs. Descriptions and diagrams of the plant are appended. H. C. R.

1311. Treating Hydrocarbon Oils with Metallic Halides. R. W. Hanna, Assr. Standard Oil Co. (Calif.). U.S.P., 1,820,389, 1,820,390, 25.8.31. Appl., 27.11.26.

A process is described in which hydrocarbon oils are contacted with metallic halides under pressure, in successive portions, the sludge formed being removed and the incompletely reacted oil recycled. It is claimed that larger yields of naphtha are obtained per unit of metallic halide. A diagram of the requisite plant is included. Care is taken to remove all oxygen and oxygen containing compounds with the first addition of halide. The process may be used as a cracking process or a polymeriser. H. C. R.

1312. Method for Cracking Oil. C. L. Smith. U.S.P., 1,822,753, 8.9.31. Appl., 25.1.29.

The object of the invention is to provide a system for cracking hydrocarbons in the vapour phase continuously and wherein provision is made for fractionating the charging stock prior to treatment in the converting zones, whereby each zone receives charging stock having relatively narrow boiling range limits. By this method optimum temperature conditions can be maintained in each converter to conform with the characteristics of the oil under treatment, thus over-cracking of lighter compounds and under-cracking of heavier constituents is avoided with reduction in gas formation and the production of a superior end product. R. P.

1313. Converting Hydrocarbon Oils. L. Burgess. U.S.P. 1,822,861, 8.9.31. Appl., 13.4.27.

This patent relates to the conversion of petroleum oils to lower b.-pt. products by heat treatment with catalytically active aluminium chloride at a temperature below that of the b.-pt. of the raw stock. The process is continuous, and means are indicated for the proper adjustment of the aluminium chloride feed to enable the equilibrium conditions to be reached in the minimum time and to be maintained indefinitely. Diagrams are provided. H. C. R.

1314. Converting Oils. N. E. Loomis and L. Burgess, Assrs. to Standard Oil Dev. Co. U.S.P. 1,822,862, 8.9.31. Appl., 7.5.27.

Hydrocarbon oils are converted into lower boiling oils by heating the former with aluminium chloride, more particularly at higher temperatures than are possible under normal atmospheric pressures. It has for its object an increase in the amount of low boiling oils recoverable, an improvement in efficiency of the aluminium chloride catalyst, and an increased output from

apparatus of any given size. The oil is heated in the presence of aluminium chloride to between 650° and 750° C., and distilled while maintaining a pressure of between 100 and 300 lb./sq. in., said pressure being controlled to maintain the desired characteristics of the converted products. W. H. T.

1315. Converting Petroleum Oil. G. Egloff. U.S.P. 1,822,924, 15.9.31. Appl., 2.6.27.

The oil is passed through a cracking coil and heated to a cracking temperature under pressure. The cracked oil is introduced into a vaporising chamber, where a substantial portion vaporises and passes to a second chamber, thence to a dephlegmator. The residual oil from the first chamber flows to a receiver, where the pressure is reduced so that a substantial portion of the oil vaporises. The vapours produced are mixed with the reflux from the second chamber and passed to a secondary heating coil; then, after the greater heat treatment, are returned to the first chamber under sufficient pressure. R. P.

1316. Soaking Drum and Method of Reconditioning the Same. J. K. Hencken. U.S.P. 1,823,451, 15.9.31. Appl., 12.11.27.

The coke collected in soaking drums employed in oil cracking has usually been removed by breaking up the mass of coke, thus leaving the chamber bare and exposed to attack by the hot liquid undergoing treatment.

This invention contemplates a soaking drum having a protective lining composed of coke. According to the invention, petroleum is passed through the soaking drum in the usual manner, with the result that the drum eventually becomes filled with a homogeneous and cohesive mass of carbonaceous material. The removal of the carbonaceous material is confined to the inner portion of the mass, and a lining of substantial thickness is left inside the drum. The coating of coke thus left is cohesive and refractory, and is not removed by the subsequent use of the drum.

The specification illustrates a machine adapted for the purpose of removing a large percentage of the coke mass, but leaves the protective lining. The machine forms no part of the patent, but is described and claimed in a co-pending application. R. P.

1317. Producing a Slurry of Lime in Oil. E. E. Bartels. U.S.P. 1,823,582, 15.9.31. Appl., 9.7.27.

The apparatus specified consists of an open tank having an air pipe situated at the bottom. Connections are made from the bottom of the tank through a pump, thence to a nozzle and back to the top of the tank. Immediately in front of the nozzle, a collar supports a hopper for supplying the lime.

In operating the process, the tank is filled with oil, which is circulated from the bottom via the pump through the nozzle back to the upper part of the tank. Powdered lime is dumped into the hopper and drawn by it into the stream of oil issuing from the nozzle and thereby carried into the tank. Air is injected to keep the oil in a state of agitation to prevent settling of the lime. After the requisite quantity of lime has been added, a suitable quantity of water is poured into the hopper and admixed with the oil, thus materially assisting in the production of a stable suspension of lime in oil.

While drawing off the oil for use the air agitation should be continued and circulation of the slurry maintained by the pump. R. P.

1318. Apparatus for Fractionating Oils. F. W. Hall. U.S.P. 1,823,897, 22.9.31. Appl., 27.1.28.

The invention has special reference to the coil and drum type of cracking plant connected to bubble towers wherein the cracked gasoline is separated from reflux condensate.

A process is claimed wherein cracking is accomplished by passing the oil under pressure through a primary and a secondary heating coil delivering the oil into an enlarged chamber, passing the vapours into a fractionating tower to form overhead distillate and a condensate, passing highly heated oil, from the heating coil before it passes into the enlarged chamber, through a coil in contact with the liquid condensate to maintain the latter at a vaporising temperature, and returning the highly heated oil through the coil to the heating zone for further heating to cracking temperature, then passing it into the cracking chamber. R. P.

1319. Producing Naphtha from Hydrocarbon Oils. M. S. Chappell. U.S.P. 1,823,967, 22.9.31. Appl., 1.12.26.

The invention relates to a process whereby cracking is effected in the presence of aluminium chloride.

The preferred continuous process is performed by passing fresh oil through a heating coil into a reaction chamber. The pressure on the reaction chamber is preferably above atmospheric though not essential. Additional heat may be applied to the reaction chamber if desired although not generally necessary. Within the reaction chamber the oil reacts with the aluminium chloride or metallic halide from the dephlegmating tower. The reaction in the reaction chamber is regulated so that all the metallic sludge is reduced to solid mass, while at the same time all the hydrocarbon oil not decomposed into coke is vaporised. The vapours pass into a dephlegmator wherein the light oils are separated from the heavier oils.

The metallic halide is added either continuously or intermittently into the dephlegmator and comes into contact with the oil in the dephlegmator. It reacts upon the oil for the further production of naphtha and polymerises any cracked stock. The metallic halide, it is understood, in its descent of the dephlegmator progressively comes in contact with the condensed oil in each compartment, and is finally collected in the lower end of the tower, thence passes to the reaction chamber. In the bottom of the dephlegmator both metallic halide sludge and high boiling oils collect. These stratify so that the hydrocarbons oil occupies the upper layer. The tower is provided with an automatic control which conducts the metallic halide into the reaction chamber whilst the oil is withdrawn from the upper layer to a receiver for processing in any desired manner. R. P.

See also Abstract No. 1337.

Hydrogenation.

1320. Pressure Hydrogenation with Iodine as Catalyst. J. Varga and L. Almási. *Brennstoff Chem.*, 1931, **12**, 327–29.

Hydrogenation experiments were carried out on 200 gm. samples of phenol, *p*-cresol, crude oils, tars and Eocene brown coal with and without iodine (as much as 0·5%), under 100–110 atm. initial pressure of hydrogen and at a temperature of 450° C. in rotary steel autoclaves. The fractions of crude oils and tars distilling above 300° C. were converted into benzine with and without the addition of iodine. In the case of coal tar, the fractions distilling above 300° C. were very resistant towards hydrogenation without iodine; with iodine the quantity of phenols produced increased on addition of iodine,

On repeating the research the authors found that different results were obtained with various crudes under the same conditions, whilst many crudes gave on a second or third hydrogenation gradually better or worse results. This was attributed to the presence of substances containing or arising from asphalt-forming substances. W. S. E. C.

1321. Treatment of Carbonaceous Materials with Hydrogen in the Presence of Catalysts. H. D. Elkington, assr. to N.V. de Bataafsche Petr. Mij. E.P. 353,506, 27.7.31. Appl. 25.4.30.

The patent concerns a process for the treatment of carbonaceous materials (coal, lignite, cellulose, petroleum, petroleum products, asphalt and various types of tars) with hydrogen at elevated temperatures and pressures in the presence of catalysts. The latter consist of metals or metal compounds, which in colloidal condition have been deposited on suitable carriers other than carbon. For example, the catalyst in a colloidal state may be adsorbed by precipitated metal hydroxide and the resultant mass finally dried.
W. H. T.

1322. Manufacture of Valuable Hydrocarbons by Destructive Hydrogenation. I.G. Farbenind. A.-G. E.P. 354,181, 28.7.31. Appl. 28.4.28 and 12.5.30.

A process is claimed for the destructive hydrogenation of hydrocarbon mixtures in the vapour phase and in the presence of catalysts. Hydrocarbon mixtures boiling between 60° and 325° C. may be treated by keeping the partial pressure, in the reaction vessel, of the improved products, which boil below 200° C., greater than 10% of the total pressure. The temperature of operation is in the neighbourhood of 500° C., or higher, under a pressure of at least 50 atm.

Claims are made for maintaining a higher partial pressure of the resulting products boiling below 200° C., the richer the initial hydrocarbon mixture is in hydrogen; also for maintaining a higher partial pressure on the resulting products boiling below 200° C. the lower the boiling point of the initial hydrocarbons; and employing a catalyst activated by an addition of Rhenium or a compound of Rhenium. R. P.

See also Abstract No. 1310.

Chemistry of Petroleum.

1323. Distillation of Ternary Heterogeneous Mixtures. J. Barbaudy. *Ann. des Comb. Liquides*, 1931, **6**, 229–242.

A study has been made of the boiling-point and miscibility relationships of the system methyl alcohol-benzene-water. The boiling point and refractive indices and densities at 25° C. were determined for the range of homogeneous mixtures. Water was then added till the system became opalescent, the operations being carried out in a thermostat at 25° C., and the results plotted on the usual triangular diagram. The region of heterogeneity is greater with methyl alcohol than with the corresponding system with ethyl alcohol. The methyl alcohol system is peculiar in showing an inversion of the benzene and aqueous layers, this occurring with the complex of about 52.5% benzene content. The density at this point is ·8700 and the benzene layer tends to form a spherical globule floating in the aqueous phase.

The aqueous solutions have a minimum density of ·8454, corresponding to 41·5% benzene, and a maximum of ·8515 at the critical point, where the composition is methyl alcohol 30·8%, benzene 65%, and water 4·2%. Corresponding variations occur in the benzene layer. Boiling points were determined for a number of heterogeneous systems.

The azeotropic mixture boils at 58·30° (about 39·5% methyl alcohol). There is no ternary azeotrope and hence water cannot be eliminated by Young's method. E. B. E.

1324. Heat of Formation of Water and the Heats of Combustion of Methane and Carbon Monoxide. A Correction. F. D. Rossini. *Bur. Stand. J. Res.*, 1931, **7**, 329–330.

In the original paper on this subject (Abstract 434), the data were reduced to a constant pressure of 1 atm. by means of the formula of Schuller & Wartha, which procedure has now been proved incorrect. A list of omissions to be made in the original paper is given, and the corrected results compared with those previously published. D. L. S.

1325. Catalytic Oxidation of Non-Benzenoid Hydrocarbons and Mineral Oils in the Vapour Phase. R. Shimose. *Sci. Papers Inst. Phys. Chem. Res.*, Tokyo, 1931, **15**, 251–76.

Maleic acid was obtained as a main product together with HCHO and CO_2 through the catalytic oxidation of n-pentane, n-heptane, n-octane, 2-amylene, 2-isoamylene, diisoamyl and cyclohexane with air in the vapour phase. Both maleic and phthalic acids were obtained as the main products together with HCHO and CO_2 through the catalytic oxidation of decahydronaphthalene with air in the vapour phase. Maleic acid, or both maleic and phthalic acids were obtained as the main products together with HCHO and CO_2 through the catalytic oxidation of the various fractions of some mineral oils, with air in the vapour phase. Maleic acid was obtained as a main product from gasoline, and both maleic and phthalic acids at the same time from kerosine, kerosine treated with 98% H_2SO_4, spindle oil treated with 98% H_2SO_4, liquid paraffin and paraffin. It is suggested that the mineral oils contain polynaphthenes which are oxidised to phthalic acid by catalytic oxidation in the vapour phase. W. S. E. C.

1326. Significant Temperatures in the Pyrolysis of Certain Pentanes and Pentenes. J. F. Norris and G. Thomson. *J.A.C.S.*, 1931, **53**, 3108–3115.

The temperatures have been determined at which n-pentane, iso-pentane, pentene-1, pentene-2 and trimethylethylene begin to decompose when heated in a Pyrex glass vessel for one hour. Determination of the rates at which these hydrocarbons decompose showed that for each the increase in rate was constant per ten degrees with certain temperature limits and that the increase was greater within higher temperature limits. It is probable that the limiting temperature of the several ranges are the temperatures at which certain bonds undergo pyrolysis. The results show that pentene-1 and pentene-2 decompose three times as rapidly as n- and iso-pentane when the hydrocarbons are heated ten degrees above their respective cracking temperatures. Trimethylethylene decomposes about twice as fast as the straight chain olefines and six times as fast as the paraffins under similar conditions, although within the same temperature range it decomposes roughly twice as fast as the pentanes. A trace of moisture, while having no effect on the cracking temperature of pentene-2, doubled the rate at which decomposition took

place. Heating the olefines at increasing temperatures, gave a definite temperature at which decomposition ceased. This indicates that at this point the expansion which results from pyrolysis is equal to the contraction due to polymerisation. S. F. B.

1327. Isomers in "Diisobutylene." F. C. Whitmore and S. N. Wrenn. *J.A.C.S.*, 1931, **53**, 3136–3142.

Careful fractionation of several samples of "diisobutylene" through improved laboratory columns of the adiabatic, total condensation, variable take-off type, has demonstrated the possibility of isolating both of the isomeric octenes present, 2·4·4–trimethylpentene-1 and 2·4·4–trimethylpentene-2, as well as "triisobutylene." The pure octenes were found to possess the following properties :—

2·4·4–trimethylpentene-1, b.p., 100·1–100·2°c/736 mm. and 101·2–101·3°c/760 mm., d_4^{20} 0·710 and n_D^{20} 1·408.

2·4·4–trimethylpentene-2, b.p., 103·0–103·4°c/736 mm. and 104·1–104·5°c/760 mm., d_4^{20} 0·715 and n_D^{20}, 1·415.

The boiling points were determined in a Cottrell apparatus and are corrected. The former is present in much the larger amount. Experiments showed that "diisobutylene" does not polymerise or depolymerise on standing in glass bottles exposed to light for over a year or on reflux at atmospheric pressure for six hours. S. F. B.

1328. Number of Isomeric Hydrocarbons of the Methane Series. H. R. Henze and C. M. Blair. *J.A.C.S.*, 1931, **53**, 3077–3085.

No direct or simple relationship appears to exist between the total number of isomeric hydrocarbons of the methane series and their carbon content. However, by separating the isomeric hydrocarbons of each specified carbon content, arbitrarily chosen upon the basis of their structural formulae, a relationship may be established between the number of hydrocarbons and the alkyl groups of which the former may be considered to be composed. Mathematical formulae of the (finite) recursion type are advanced which permit of the calculation, from their carbon content, of the number of isomeric hydrocarbons in each of these structural types. In using these formulae the total number of alkyl groups (*i.e.*, the total number of isomeric alcohols) of N/2 (where N is the carbon content) and all lesser carbon contents must be known. The totals obtained by means of these formulae agree with the number of tetradecanes required by theory as tested by actually writing all the possible forms. S. F. B.

1329. Condensation of Hydrocarbons by Electrical Discharge VIII. —Condensation as a Function of Time and Pressure. S. C. Lind and G. R. Schultze. *J.A.C.S.*, 1931, **53**, 3355–3365.

A large series of general principles has been described which shows the influence of pressure and time on the condensation of methane, ethylene and acetylene, and in addition the effect of the electrical discharge on the liquid products has been studied. In contrast to the reaction under the influence of alpha-radiation, acetylene is hydrogenated to some extent in the electrical discharge. W. H. T.

1330. Action of Radon on Some Unsaturated Hydrocarbons. G. B. Heisig. *J.A.C.S.*, 1931, **53**, 3245–3263.

Determinations have been made of the ratios of the number of molecules of hydrocarbon reacting per ion pair formed (—M/N) on methylacetylene, dimethylacetylene, butene and isoprene. These compounds were purified and mixed with radon in an apparatus described by Lind and Bordwell,

the number of ions produced by the radon being calculated by a modification of Mund's method. With the exception of isoprene, the values were shown to be constant throughout the course of the reaction, while the ratios were seen to be additive and constitutive. The lower the ratio

$$\frac{\Delta\,(H_2+CH_4)}{-\Delta\,H.C.,}$$

the higher is the $-M/N$ ratio, the values of the latter being compatible with the ion cluster theory. W. H. T.

1331. Transformation of Benzene into Methane. A. Maillo and Creusot. *Compt. rend.*, 1931, **193**, 60–63.

A quantitative study of the formation of methane on passing benzene and hydrogen over heated nickel showed that the yield: (1) depended on the mode of preparation of the catalyst; (2) was highest when the benzene was free from thiophen; and (3) commenced below 250°, reached a maximum at 280°, then falling until 310°, and subsequently rising (sometimes to a maximum). The activity of the catalyst decreases with time at 280°, owing to deposition of a yellow substance, soluble in acetone, but is restored by raising the temperature to 320–330°, the yellow substance then disappearing. The maximum yield obtained was 82·3%. W. S. E. C.

1332. Hydrocarbons as Dispersion Media. B. C. Soyenkoff. *J. Phys. Chem.*, 1931, **35**, 2993–3010.

The literature relative to the preparation of benzenesols and their behaviour in the electric field is reviewed, and some additional experiments are described, while electrical factors of stability are shown to be unimportant in hydrocarbons as dispersing media. W. H. T.

1333. Catalytic Action of an Aluminium Oxide Catalyst. W. D. Bancroft and A. B. George. *J. Phys. Chem.*, 1931, **35**, 2943–9.

It is shown that the increase in the relative amount of ethylene obtained by sintering an aluminium catalyst is considerably less than the difference between the dehydrating and decarboxylating catalysts. The catalysts prepared by heating $Al(NO_3)_3{\cdot}9H_2O$ favour the formation of larger amounts of ethylene than do those prepared by the precipitation with ammonium hydroxide. The use of washed pumice as a catalyst gives very large amounts of ethylene with little carbon dioxide. While there was a slight increase in the amount of ethylene obtained by sintering a catalyst, it was small compared with the effect of adsorbed substances. The work indicates that Taylor's theory, which states that the extent of the changes induced in the adsorbed reactant will be determined by the specific individual catalytic activities of the adsorbed ions or their reaction products, is probably the correct one. W. H. T.

1334. Synthesis of Aromatic Compounds at High Pressures. G. T. Morgan and D. D. Pratt. E.P. 353,464, 20.7.31. Appl., 20.3.30.

A process is described for the synthetic production of organic compounds. Carbon dioxide is caused to react with aromatic hydroxy or amino bodies in the presence of metallic halides of the type adapted to promote the Freidel and Crafts reaction under conditions of high pressure and moderate or elevated temperature. W. H. T.

1335. Carrying Out Chemical Reactions with the Aid of Electric Discharges. J. Y. Johnson, Assr. to I. G. Farbenind A.-G. E.P. 354,735, 5.8.31. Appl., 5.2.30.

The patent concerns improvements in carrying out chemical reactions with gases, vapours or mists of solid or liquid substances with the aid of electrical discharges. The current of the said substances is freed from secondary vortices by passing it through a cylindrical or slightly conical reaction space through which passes an electric discharge, the current being led to this space through an annular passage coaxial therewith. The passage has smooth unbroken surfaces which form a right angle with the axis of the discharge and which lead the current thereto. The surfaces have an area which is at least ten times as large as the cross-sectional area of the reaction space. W. H. T.

Analysis and Testing.

1336. Spectral Changes which Accompany Deterioration of Mineral Oils. M. E. Vellinger. *Ann. des Comb. Liquides*, 1931, **6**, 225–228.

The speed of deterioration of mineral oils can be followed by means of a spectrophotometer (cf. Abs. 814A, and *Ann. des Comb. Liquides*, 1930, **5**, 273–282). The deteriorated oil is dissolved in a colourless solvent, *e.g.*, a refined mineral oil of Russian or American origin, and it is found that both Beer's and Lambert's laws apply, viz. :—

Lambert. $\log \frac{I}{I_0} \propto l$, for constant concentration.

Beer. $\log \frac{I}{I_0} \propto c$ for constant length.

— where I_0 = intensity of entering light, visible spectrum.
I = intensity of light leaving sample.
l = length of oil traversed by light (1 to 25 cms. in these experiments).
c = amount of deteriorated oil present in solution.

Hence the extent to which deterioration has occurred can be followed. W. E. J. B.

1337. Laboratory Control of Cracking Still Charging Stock has Reduced Formation of Coke. A. W. Trusty. *Oil and Gas J.*, 1931, **30**, 20-21.

The selection of suitable crudes for topping operations is relatively simple, being effected by a laboratory distillation followed by examination of the fractions taken off. The degree and cost of refining necessary can also easily be determined without carrying out an actual plant run, while the anti-knock quality of the gasoline can be ascertained from its octane number.

The evaluation of charging stocks for cracking is much more difficult. The most important tests are :—(1) Tar number ; (2) Aniline number ; (3) Distillation tests.

(1) *Tar number.*—This gives an indication of the carbon-forming compounds in the cracking stock, and thus the tendency for coke formation. It is determined by agitating 100 c.c. of oil with 10 c.c. sulphuric acid (60° Bé)

and measuring the residue after standing. With a heavy fuel a mixture of 50 c.c. oil and 50 c.c. kerosine is used. Tar numbers for various stocks are: Gas Oil (30–36° Bé) 2–4; East Texas Crude (39° Bé), 16; Residue from East Texas Crude (26° Bé), 26; Fuel Oil (14° Bé), 100.

(2) *Aniline number.*—This gives an indication of the chemical composition of the stock. It is determined by noting the lowest temperature at which 10 c.c. of freshly distilled aniline and 10 c.c. of the oil to be tested are just completely miscible. A high aniline number indicates a paraffinic stock which cracks relatively easily, but produces a gasoline with a comparatively poor anti-knock value. A low aniline number indicates a naphthenic or aromatic stock which must be cracked at a higher temperature (or with longer time) but yields comparatively good anti-knock gasoline.

The character of the cracking stock is of much more importance, in liquid phase cracking, in its bearing on the resulting product, than it is in vapour phase cracking. In cracking a paraffin stock symmetrical cracking of the molecule with no coke results. The coke which is formed at high temperature is due to secondary cracking which is accompanied by an increase in olefines, methane and hydrogen leading to formation of diolefines, then aromatics and finally coke.

Recycling of the charging stock results in a reduction of the aniline number of the recycle oil and an increase of the amount of gas formed.

(3) *Distillation tests.*—The more closely fractionated the charging stock, the more efficient is the cracking, owing to the fact that with wide cut charging stock at the temperature necessary to crack the lightest fractions, the heaviest fractions are over cracked, while at the temperature necessary to crack the latter the former will be practically unaffected. The rate of cracking is a function of the boiling range of the cracking stock, light gas oil taking nearly twice as long to crack as heavy fuel and kerosine five times as long as gas oil. It is concluded that the ideal cracking stock is a closely fractioned heavy gas oil with a low tar number and a low aniline number. C. L. G.

Anti-Detonation.

1338. Peroxides in Gases of Explosion Motors. P. Dumanois, P. Mondain-Monval, and B. Quanquin., *Ann. des. Comb. Liquides.* 1931, **6**, 559-562.

It has already been shown (cf. Abstract 694) that alkyl peroxides are formed during the direct oxidation of saturated hydrocarbons by air at a temperature of between 200 and 300° C. The time available for the formation of peroxides in these experiments was several seconds, so it was necessary to investigate the formation of peroxides in the time available during the compression stroke of an I.C. engine, viz., 0·02 sec. (approx.). A mixture of air and hexane was examined in an air-cooled, single-cyl, 4-str. engine, of 70 mm. bore and 70 mm. stroke. The engine was run normally at about 1200 r.p.m., with arrangements to absorb the power, and the increase in temperature of the cylinder, which was insulated with asbestos, was observed. Around 270° C. detonation became apparent, so the ignition was switched off and the engine motored at the same speed by means of an auxiliary motor. Samples of exhaust gases showed the presence of peroxides and aldehydes, and their condensation and decomposition products. Moreover, on switching off the ignition, slight explosions were heard, and since no flame could be

observed through a thick glass window in the cylinder head, the authors considered that these slight explosions were caused by the exothermic and spontaneous decomposition of the peroxides formed during compression. Prior to detonation, such a test showed that aldehydes and peroxides were not present in the gaseous compression mixture. W. E. J. B.

1339. Knocking in Internal-Combustion Engines. K. Schnauffer. *D.V.L. Jahrbuch*, 1931, 375-378.

Work has been carried out at the German Institute for Aeronautical Research on the combustion phenomena occurring in engine cylinders. The pressure indicator used has been previously described (*D.V.L. Jahrbuch*, 1930). For measuring the speed of flame-travel, four spark plugs are fitted at intervals in the combustion head. The first is used for ignition; across the other three are potentials which, when the flame-front reaches the electrodes and the ionised gas-particles allow a current to pass, cause deflections in an oscillograph. It is shown that in normal combustion the flame-speed is constant across the cylinder, but when knocking occurs, the speed which has been normal up till then suddenly becomes exceedingly great, there being no measurable interval between the arrival of the flame-front at the last two plugs. The region in which knocking occurs can be measured by varying the position of the plugs: in some cases it is 50% or more. From the time-pressure diagrams it is shown that before knocking a period of delayed combustion occurs, compared with normal burning. The combustion, however, is then rapid and complete, no after-burning occurring as in the case of non-knocking combustion. It is not a matter of indifference at which part of the cylinder the pressure-indicator is placed: this should be in the knocking zone, and not near the spark plug. In some cases a negative flame-speed was observed, due to very rapid expansion of the portion of the charge igniting instantaneously. Knocking is divided into four classes: the normal type, with delayed burning and then a perpendicular pressure rise; the pre-ignition type; the "rough running" (Ricardo) type, due to excessive turbulence; and a double flame-front type, due to ignition from the overheated walls as well as from the spark. E. B. E.

1340. Mechanism of Combustion in Engines and the Six-Cycle Principle. E. Terres. *Z. angew. Chem.* 1931, **44**, 509-19.

Photographic records have been made of the rate of flame propagation in explosive mixtures, tests being made on mixtures of air with C_6H_6, PhMe, and $C_6H_3Me_3$ with varying air-fuel ratio, initial pressure and initial temperature. It was found that the rate of flame propagation increases with initial pressure up to about 15 atm. after which it decreases; wet mixtures burn faster than dry mixtures, this suggesting an explanation for the decreasing of the rate of combustion by preheating. The results point to the advantages of the 6-cycle principle in which a rich mixture is first exploded and a secondary combustion is started after the admixture of the remainder of the theoretical amount of air for complete combustion. W. H. T.

1341. Photographic Analysis of Explosion Flames. W. A. Bone. *Nature*, 1931, **128**, 589.

A survey is given of the method devised by Mallard & Le Chatelier for the photographic analysis of explosion flames, which was later developed by Dixon. Further improvements have been recently made by the use of Fraser's high-speed photographic machine whereby photographs and measurements of movements in explosive flames occurring with a frequency of 250,000/sec. can be made. It is hoped to obtain further analysing power

by a camera in which a mirror revolving in vacuo at constant velocity (16,000 r.p.m.) projects the image of the explosion flame on a stationary film. Probably Mallard & Le Chatelier's suggestion of flame propagation by conduction is an ideal condition. It has been proved that the speed can be abruptly raised from a lower to a higher uniform value, when a flame is overtaken by a "shock" wave travelling in the same direction. During the predetonation phase of explosion, the flame is advancing at a speed greater than that of any shock wave through the unburnt medium. When a flame on the point of detonation is about to overtake a shock wave immediately in front of it, an ignition ahead of the flame front takes place, followed by detonation.

The phenomenon of spin has been observed in detonations of methane-oxygen, pentane-oxygen, undiluted acetylene, etc. It is apparently caused by the helical rotation of a luminous head of detonation in the flame front, together with a long luminous "tail" behind it. C. C.

Engines.

1342. Prevention of Valve Seat Erosion. E. M. Getzoff. *J.S.A.E.*, 29, 332–335.

The Plainfield Laboratory of the International Motor Co. have developed a special alloy for valve seat inserts, known as "Mack Alloy." Details of the alloy are not given, beyond stating that a high percentage of tungsten is employed. A theory of the cause of the familiar hard, spotty formation between valves and cast iron seats is advanced, and it is pointed out that this condition does not obtain with non-ferrous insert seats. The troubles experienced when using aluminium bronze inserts are detailed, such as that of permanent fixing due to high coefficient of expansion, and several methods of fixing to cylinder block, which are more or less satisfactory are shown. The Mack Alloy inserts employ a flat seat, and details of prolonged tests in the laboratory and the field are given which appear to be highly satisfactory. The recession of valve seats of Mack Alloy after 500 hr. test, are shown to be only one-seventh that of aluminium-bronze seats and one-twenty-fifth of that experienced with cast-iron. A special method of fixing is shown which is claimed to be quite satisfactory and easily carried out without special tools. C. H. S.

1343. Heating Charges Supplied to I.C. Engines. G. R. Welch. E.P. 353,714, 30.7.31. Appl., 1.8.30.

The invention provides means for heating the petrol air mixture supplied to I.C. engines by utilising the heat of the exhaust gases. A hollow body is used as a valve which connects a chamber in the exhaust manifold to the induction manifold. This chamber is always hot when the engine is running, so that the mixture is heated as soon as the valve is turned. The latter being hollow acts as an insulator to prevent heating of the mixture when the hot chamber is not communicating with the induction manifold. J. M. F.

1344. Liquid Fuel Injection Pumps for I.C. Engines. D. Napier and Sons, Ltd., R. W. Vigers and E. E. Chatterton. E.P. 353,771, 30.7.31. Appl., 26.9.30.

The invention relates particularly to that type of liquid fuel pumps in which the pump piston acts both as plunger and as the valve controlling the inlet or relief ports. It provides means for positively closing the delivery

passage at the moment when, or immediately after, the relief port is opened to terminate the delivery stroke. Thus the earlier and later portions of the plunger delivery stroke are inoperative, and fuel is only forced through the delivery port during an intermediate operative part of each delivery stroke.
J. M. F.

See also Abstracts Nos. 1278, 1279.

Coal and Shale.

1345. Use of Primary Tars from Coal. J. Sainte-Claire Deville. *Ann. des Comb. Liquides*, 1931, **6**, 189–194.

The problem of the economical exploitation of low temperature tar has been developed along two lines: (1) By one or more fractionations followed by refining, giving the usual range of products—petrol, solvents, gas oil, lubricating oils, etc.; (2) by cracking the residue (after removal of the petrol fraction) to convert the whole to a light spirit.

The objections to the latter method are indicated—*e.g.*, the low hydrogen content of the residue giving rise to high production of coke, and the many different types of compound present which require different conditions for decomposition. Attempts have also been made to use a combined cracking and hydrogenation process; but, here again, different temperature and different catalysts are required to decompose the various types of compound.

By the first method valuable products other than petrol are obtained, but the oxygen-containing compounds (phenols, etc.) prevent it competing with petroleum residues. Later developments may use the phenolic compounds for insecticides, etc.

The main difficulty is that economical working can only be obtained on a large plant. Central refineries should be established at which not only fractionating plant should be erected, but all the subsidiary plant necessary to produce the maximum yield of commercial products.
C. C.

1346. Production of Oil from Coal. L. J. Rogers. *Chem. Age*, 1931, **25**, 269.

The history of and the chemical reaction involved in the Bergius process for the hydrogenation of coal are outlined, and the semi-technical plants installed are enumerated. Although the main function of the ferric-oxide is to combine with the sulphur in the coal, it also exerts a catalytic action believed to consist in promoting cracking mainly, but also assisting in the addition of hydrogen to unsaturated compounds. Probably the constituents of the ash also have a catalytic effect. Other catalysts are mentioned and reference made to the work of Fischer and Waterman. At Kaiser Wilhelm Institute work has been carried out on the relative values of molecular and nascent hydrogen. Substances which have been found more effective than gaseous hydrogen are mentioned. The cheapest and most effective method of treatment may combine the use of water gas in the preliminary stages and of gaseous hydrogen at the end of the process. Typical yields of products are given and their values discussed. The usage of hydrogen in the process, the quantity required in treatment and the methods devised to recover hydrogen from residual and other gases are stated. In hydrogenating coal

it may be economical to convert the residual gas into liquid hydrocarbons as far as possible, and to use the uncondensed hydrocarbons for the production of hydrogen. The separation of oils from the crude product is difficult, and so far the most satisfactory method found is to centrifuge at a temperature of about 100° C. with subsequent distillation. The methods of production of hydrogen by the "steam-iron" process, from water gas by catalytic action and by fractional liquefaction of coke-oven gas, are described.

R. A. E.

1347. Thickening of Wash Oil. R. A. R. *Gas World* (*Coking Section*), 1931, **95**, 16–17.

The amount of wash oil (creosote) used for extraction of crude benzole from gas varies from 0.1 to 0.25 gal. of oil per gal. of crude benzole. The thickening of wash oil is due to increase in viscosity and molecular weight resulting in a lowering in efficiency as regards benzole extraction. The former affects the diffusive power of the oil in the scrubbers, and the latter affects the absorptive capacity, which point is illustrated by data given. The main causes of the thickening of wash oil are: (1) Removal of lower boiling hydrocarbons by distillation in the stripping still; (2) absorption of tar fog by the wash oil; (3) polymerisation and cracking of the wash oil; (4) absorption of sulphur compounds. Suggested remedies are: better fractionation in the stripping still, removal of tar fog by electrical means, and removal of sulphuretted hydrogen from the gas before benzole extraction. The prevention of polymerisation is apparently difficult. W. A. W.

1348. Low Temperature Carbonisation and Distillation of Coal, Lignite and Oil Shale. "Intertrust" Cie Generale de Distillation, S.A., Assrs. of P. Brenn. E.P. 351,552, 29.6.31. Appl., 27.3.30.

This invention relates to a plant for the carbonisation of coal and the like in which means are provided for the ready removal of the retorts to facilitate discharge and also to preheat fresh charges of raw stock. H. C. R.

1349. Gaseous and Liquid Fuels. Ruhrchemie A.-G. E.P. 354,215, 27.7.31. Appl., 27.1.30, Conv. (Ger.), 26.1.30.

Coal or other carbonaceous solid fuels are gasified in water-gas generators. The gas produced is then subjected to a short heat treatment at a temperature exceeding 1000° C. A good illuminating gas is produced, together with an anti-knock hydrocarbon motor fuel. It is claimed that complete conversion of the raw material is obtained. H. C. R.

1350. Distilling Shale or Other Oleiferous Minerals. H. Werner. E.P. 354,962, 20.8.31. Appl., 14.7.30.

Usually minerals are introduced into retorts in a loose granular condition and the oil vapours rise quickly through the spaces and are liable to be overheated. In the present invention the material is introduced into the retort in a graded state so that fine grains will fill up the spaces between the coarser particles. With the material in this condition the charge is compressed so that the penetration of heat into the charge will be retarded. Thus the oil vapours will be able to escape without being over heated. To enable the vapours to escape easily, channels may be made by packing the material around withdrawable cores. To prevent the oil vapours from contacting with the walls of the retort, a hood connected to an outlet pipe may be placed on top of the charge inside the retort. R. P.

1351. Apparatus for Low Temperature Distillation or Other Heat Treatment of Bituminous Materials. I.G. Farbenind. A.-G. E.P. 355,020, 20.8.31. Appl., 12.8.30.

In the continuous drying and low temperature distillation of materials it frequently happens that the water vapour escaping from the lower portions of the material condenses on the upper portions causing a great tendency to cake. It is proposed to use movable bars or chains in the upper parts of the chambers, extending to a depth at which the devices are not attacked by the materials undergoing drying or decomposition. By means of these devices the caking of the charge is prevented and continuous running assured. R. P.

1352. Recovery of Soluble Products from Solid Carbonizable Substances. J. Jannek and O. Göhre, assrs. to I.G. Farbenind A.-G. U.S.P. 1,822,349, 8.9.31. Appl., 15.3.28.

A process is described in which carbonizable substances are treated by extraction with a distillable liquid organic solvent at an elevated temperature and at a pressure exceeding two atm. The raw material is previously heated to a temperature of between 100° C. and 600° C. and at a pressure of 10–100 atm. H. C. R.

1353. Purification of Tar Acid Bearing Oils. S. Caplan, assr. to Combustion Utilities Corpn. U.S.P. 1,822,842, 8.9.31. Appl., 22.9.26.

A process of extraction of the colour-producing compounds from tar oils is described. The oils are agitated with aqueous borax solution, which removes these unwanted constitutents without removing any tar acids of high germicidal value. Subsequent settling and separation yields the purified oil. H. C. R.

1354. Conversion of Solid Fuels. C. Müller, G. Wietzel and E. Galle, assrs. to Standard I.G. Co. U.S.P. 1,823,116, 15.9.31. Appl., 22.11.26.

The invention relates to the preliminary treatment of moist solid carbonizable fuels before destructive hydrogenation. The water is removed by mixing the fuels with distillates obtained from the destructive hydrogenation of similar fuels and subjecting the mixture so formed to a pressure of at least 50 atm. Details of the form of press used are appended. H. C. R.

1355. Purification of Tar Acid Bearing Oils. S. P. Burke, assr. to Combustion Utilities Corpn. U.S.P. 1,823,558, 15.9.31. Appl., 22.9.26.

The object of this invention is to remove the colour-producing compounds from the oils obtained by the distillation of bituminous materials. These compounds are said to be homologues of catechol, *e.g.*, 3·4 and 3·6 dimethyl catechol. They are removed by agitation with an aqueous solution of a non-acidic salt of a heavy metal, separation of the aqueous layer and subsequent filtration serving to remove the insoluble metal salt. Antimonyl and lead salts are particularised. H. C. R.

See also Abstract No. 1300.

General.

1356. Corrosion-Prevention. L. C. Milburn. *J.S.A.E.*, 1931, **29**, 148–157.

The paper deals mainly with the means for protecting aluminium from corrosion, under conditions which vary, from immersion in sea water (immersion of flying-boat hulls in sea water) to those in joints between dissimilar metals. The methods commented on are protective coatings—paints, oils, greases, etc., and full heat treatment, the last-named being very necessary to obtain the best corrosion resistance of the most common aluminium alloy, 17ST. C. I. K.

1357. Hydrocarbon Vapour Lamp. F. C. Tilley. E.P. 355,869, 3.9.31. Appl., 21.6.30.

The lamp concerned is one in which the vaporising tube leading from the reservoir is vertical and carries the burner and mantle at its upper end, and a preheating device near its base which carries a wick surrounding the vaporising tube. The preheater heats the vaporising tube sufficiently to vaporise the oil forced up the tube before it is ignited at the mantle, after which the heat of the burner and mantle is communicated to the tube for vaporising purposes.

The preheating device is improved by means of a valved by-pass with a supply tube to receive a preliminary supply of oil for preheating the vaporising tube. The whole device, including the by-pass pipe, can be readily removed from the vaporising tube, and the U-shaped perforated bar on the sleeve on the oil trough can be raised to light the wick. W. S. E. C.

ABSTRACTS.

Geology.

Donaldson Bogart Dowling Memorial Symposium on Stratigraphy of Plains of Southern Alberta.

The following fourteen papers were published in the October, 1931, issue of the *Bull. Amer. Assoc. Petr. Geol.*

1358. Stratigraphic Correlation and Nomenclature in Plains of Southern Alberta. J. S. Irwin. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1129–1139.

The author suggests a simplified nomenclature for all the formations above the Madison Limestone (Mississippian) up to the Upper Cretaceous to replace the large number of local names now in use. As the formations found in Northern Montana are very similar to those in the plains of Southern Alberta, the nomenclature is applicable to the whole area.

A correlation between the formations in Montana and those in Alberta is attempted. This is carried out with copious reference to other writers, and it is on the final result that the nomenclature is based. The basis of the correlation is primarily lithologic, but the fact is recognised that the lines of lithology traverse the time lines, only the author believes it better to correlate something tangible rather than the intangible. G. R.

1359. Palæozoic of Southern Plains of Alberta. P. D. Moore. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1141–1155.

In this paper the Palæozoic formations, Madison Limestone, Threeforks and Jefferson are discussed. A correlation is attempted over a considerable area from the records of seventeen wells that penetrate these formations.

There seems to be a very consistent, widespread black shale at the junction of the Threeforks and the Madison Limestone, and this is taken as a datum. It contains the fossil *Lingula spatulata*. Of the two unconformities found in Montana at the base of the Pennsylvanian and at the base of the Jurassic, the Jurassic one only is found in Alberta.

The chief difficulty found in the correlation was the great lithologic change, particularly in the Threeforks Anhydrite formation. G. R.

1360. An Ellis (Upper Jurassic) Section at East Butte, Sweetgrass Hills, Montana. J. O. G. Sanderson. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1157–1160.

There is no exposure of the Ellis formation in the plains of Southern Alberta. The nearest one is that described in this paper.

In the Sweetgrass Hills, Igneous activity has considerably metamorphosed the rocks, and this section is the only undisturbed one available. A complete description and measured columnar section are given. It is for the most part a dark shale series with medium grained sandstones, a limestone zone, some good fossil horizons and has oil showings near the base. G. R.

1361. Stratigraphy of the Red Coulee Oil Field. W. S. Yarwood. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1161–1170.

In this field, situated near the International Boundary, the Milk River Sandstone is exposed under the drift covering. Successively below are the Marine Colorado Shale (U. Cret.), the Blairmore (L. Cret. = Kootenai), the Ellis (Jurassic) and the Madison (Mississippian). Each of these formations is described with their distinctive marker beds, and the producing Blairmore formation is described and zoned in detail. G. R.

1362. Stratigraphy of Colorado Shale in the Southern Plains of Alberta. J. G. Spratt. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1171–1179.

An attempt is made to correlate the Colorado Shale which has been well studied in the Sweetgrass Arch, northward to Bow River.

Little change is shown until north of Bow River, where the Milk River Sandstone wedges out. The Colorado Shale is mainly a shale formation with occasional sandstones and Bentonitic zones. The Medicine Hat Gas Sand, previously correlated with the Milk River Sandstone, is shown to be a lenticular sandstone within the shale and about 300 ft. below this formation. The Blackleaf member found in Montana is recognisable in Alberta and contains the massive Bow Island Gas Sand. G. R.

1363. Stratigraphy of Foremost, Pakowki and Milk River Formations of Southern Plains of Alberta. S. E. Slipper and H. M. Hunter. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1181–1196.

The Foremost formation is a brackish water series deposited during a regression of the lower Montana sea. It shows three divisions: (*a*) Verdigris Sandstone (lower) representing the beach sands; (*b*) very variable, lenticular, lagoonal deposits (middle and upper) with chemical precipitates; (*c*) Pale beds, deltaic deposits with coals. The deposition seems to have been from the south-west.

The Pakowki formation is a series of marine shales and sandstones underlying the Foremost. An Isopach map shows rapid thickening towards the south-east. In the east it is mainly a shale formation passing into a sandstone phase with some shales in the south-west. It was evidently a deposit formed during a rapid transgression of the lower Montana sea.

Underlying the Pakowki is the Milk River formation, again brackish water to marine and probably formed during the retreat of the Colorado sea. The same three stages as in the Foremost are recognisable, *viz.*, a strong beach deposit, the lower Virgelle sandstone, a carbonaceous shale or lagoonal series in the middle and pebble beds as the deltaic deposits at the top. The last named is not as well defined as in the Foremost. G. R.

1364. Subsurface Study of Pale Beds and Foremost Formation in the Lethbridge-Brooks Area of Southern Alberta. D. L. Powers. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1197–1213.

This paper discusses the Foremost formation and the Pale beds from the information of seven deep wells drilled in the above area. It corroborates the work described in the previous paper by Slipper and Hunter.

The Foremost, thickness 400 ft., consists of brackish water, dark grey shales with subordinate sandstones, carbonaceous shales and oyster beds. The McKay coal series is found near the base and the Taber near the top.

The Pale beds are fresh water deltaic deposits, mostly sandstones with some shales and the Lethbridge coal series near the top. It is generally about 700 ft. thick. The author also concludes a western landmass as the source of sedimentation. G. R.

1365. Sediments of Montana Age in Milk River Edge Region, Alberta. B. F. Hake and Carl C. Addison. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1215–1225.

In this paper the authors describe the Foremost formation and Pale beds, corroborating the evidence of the two previous papers. The overlying Bearpaw Shales are about 700 ft. thick and are mainly a shale formation with sandstone phases, calcareous nodules and some bentonitic bands.

The authors also conclude a regression of the Montana sea as the history of the Foremost, but point out that it was an oscillatory movement. At the end of the deposition of the Pale beds the terrain was remarkably flat and the drainage of little power. The subsequent Bearpaw represents a long period of quiet marine sedimentation, and they therefore conclude sudden subsidence at its inauguration with little or no beach deposit to commence with. G. R.

1366. Bearpaw Shale and Contiguous Formations in Lethbridge Area, Alberta. T. A. Link and A. J. Childerhose. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1227–1242.

The Pale beds graduate upwards into the Bearpaw Shale passing through the Lethbridge coal series, some of which are of economic importance. The Bearpaw shale formation has a middle sandy phase and shows many bentonitic bands, some of which are consistent and useful for mapping. The upper part is also a sandy formation with some shale, there being three distinct sandstones.

Overlying the Bearpaw is the Fox Hills Sandstone, being a continuous massive sandstone 140 ft. thick with a break of carbonaceous shale near the top. The author, contrary to Hake and Addison (see previous paper), concludes an advancing sea as depositing the Bearpaw, the lagoonal Lethbridge being a continuous sheet younger on the north-west than on the south-east. The Bearpaw graduates upward into the Fox Hills Sandstone indicating a gradually withdrawing sea, and thus the Fox Hills is also an overlap sheet. G. R.

1367. Sections of Bearpaw Shale from Keho Lake to Bassano, Southern Alberta. C. M. Clark. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1243–1249.

The description of the Bearpaw Shale given here confirms the remarks of the previous authors in this symposium. The three lithological units are noted, *viz.*, a basal shale member with bentonitic beds and no sand, a middle sandstone group, usually fine-grained and fairly massive, and an upper sandy group with lenticular sandstones and sandy shale. The author concludes a marine deposition oscillating slightly from deep water to off-shore conditions, but with a general tendency towards the latter until it ends up with definite off-shore conditions depositing the Fox Hills Sandstone. G. R.

1368. Fox Hills Formation in Southern Alberta. J. O. G. Sanderson. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1251–1263.

The Fox Hills is an arenaceous formation of littoral marine type, giving place to Estuarine at the top where it becomes the first St. Mary River Coal. The source was dominantly the western uplands and it was deposited slowly with little current bedding and relatively complete sorting. A distinctive fauna is given and stratigraphical descriptions of the formation in many localities. G. R.

1369. Stratigraphy of Spring Coulee Well. W. S. Yarwood. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1265–1277.

This paper is detailed description of the formations passed through by a single well, depth 5970 ft. They were: Drift; St. Mary River (Estuarine); Marine Fox Hills Sandstone; Marine Bearpaw Shale; Belly River group (Pale beds, Foremost Pakowki, etc.); Marine Colorado Shale; Blairmore formation; Marine Fernie Shale; and Upper Palæozoic Limestone. Correlations are suggested. G. R.

1370. Stratigraphy of Spring Coulee Well, Supplementary Paper. W. P. Mellen and F. W. Rohwer. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1279–1282.

This paper continues the log of the Spring Coulee well down to 6,191 ft., well into the Madison Limestone. Detailed description is given of the log. Considerable dolomitisation and secondary silicification is shown, the latter particularly at the upper and lower zones. The Limestone here is believed to be considerably younger than in the plains well to the north and east. G. R.

1371. Invertebrate Palæontology of Southern Plains of Alberta. P. S. Warren. *Bull. Amer. Assoc. Petr. Geol.*, 1931, **15**, 1283–1291.

This paper is a description of the faunas of the Cretaceous rocks found in this region from the Milk River sandstone upwards. Certain forms can be ascribed to certain horizons, but generally the faunas are fairly constant throughout Montana time. G. R.

1372. Possible Oil and Gas Field in North-Western Florida. J. F. Weinzierl. *Oil Weekly*, 1931, **63** (5), 58.

The existence of a considerable subsurface uplift on the western flanks of the major uplifted area of Florida, as indicated by a widespread geophysical survey over north-western Florida in 1930, has been proved by a well located 8 miles south of De Funiak Springs in Walton County, Florida.

The well penetrated 3105 ft. of Tertiary beds, encountering gas shows at 375 ft. and 1177 ft., and oil and gas shows at 2615 ft. and 3050 ft., the latter being most promising. The well is now drilling at 3400 ft. in the Selma Chalk division of the Upper Cretaceous and should reach the Eutaw formation, the sandy portion of which showed oil in Western Alabama, at 3850 ft. The well shows a much greater percentage of sand and carbonaceous shale in its section than has been found in wells drilled further to the East, where lime formations predominate. C. E. H.

Field Technology.

1373. Drilling Under Pressure in California. B. Mills. *Oil Weekly*, 1931, **63** (6) 14.

Drilling under pressure has solved many conditions which have baffled operators in the past. Such conditions as high water pressure, heaving formations, and the loss of fluid under unusual conditions can be adequately countered.

A deep test in the Lost Hills Field has produced some interesting facts regarding this system. Special pressure equipment was used, and the drilling of 2000 ft. under this arrangement was accomplished only after a series of problems had been met and solved. In this well both water pressure and heaving formations added to the difficulties.

G. F. Getty's No. 1 Well was drilled to a depth of 1545 ft., where it encountered a strong gas pressure. By weighting the mud the well was drilled to 1845 ft., but the drilling speed was considerably reduced between these two depths owing to heaving shale and heavy gas pressure. Admixtures were added to the mud to overcome the heaving condition, but these were not completely successful. A blow-out preventer was then installed and a pressure of 400 lb applied to the hole. As a result of this, circulation was made at points around the rig. After attempts had been made to seal these crevices the well was abandoned.

A second well was then started and was drilled with ordinary rotary equipment to 1098 ft., when circulation was lost. A string of 18⅝-in. casing was cemented at 1000 ft. in order to eliminate the possibility of danger to the rig foundations and establish circulation. Circulation was regained and drilling continued to 1371 ft., where heaving shale and gas pressure gave trouble. By increasing the weight of the mud to 125 lb the well was drilled to 1,870 ft., where circulation was again lost. It was then found that the mud was coming up on the outside of the 18⅝-in. casing and in the original hole. After drilling to 1921 ft. a string of 11¾-in. casing was run in and landed at 1916 ft. Drilling was then continued to 2068 ft. by using the 125-lb mud, but here a water sand with a pressure of 1600 lb was encountered.

At this point pressure equipment was installed and drilling resumed with 85 lb drilling mud. A gas control and flush joint drill pipe was used and 600 to 625 lb pressure was needed to keep back the salt water. By maintaining a pressure slightly higher than 625 lb the water pressure was overcome with the loss of from 50 to 100 brls. of mud daily. A pressure of about 650 lb plus the hydrostatic pressure of the head of the fluid in the well was maintained, and this prevented bridging of the sand.

When going into the hole with the 5-in. O.D. flush-joint drill-pipe it was necessary to pull or force in the first few stands with the snubbing and pull down clamps. After a few joints had been run the weight of the drill-pipe was sufficient to overcome the pressure maintained in the hole and the drill-pipe dropped without the use of the device. In coming out of the hole the upper section was pulled, but it was necessary to reverse the action and hold back the last few joints with the snubbing device to prevent them from being blown from the hole.

Drilling was continued in this way until a depth of 3700 ft. was reached, when continuous coring was started. The mud had been reduced to 72–75 lb. While taking the last core between 3753 and 3768 ft. the gauge recording the hole pressure jumped from 650 to 850 lb. The mud thinned out rapidly and the pressure continued to rise until it reached 1300 lb. Cement in large quantities was pumped into the well without success, and sacks of weighting compound also failed to have any effect. It was assumed that the mixture was being jetted out through the water courses at 2068 ft., where a water-pressure of 1600 lb was encountered. Since the mixture was being forced into the formation at 2068 ft. it was obvious that the pressure at 3764 ft. must have been greater.

The cementing of the string of 8¼-in flush-joint casing was expected to make deeper drilling possible under more favourable circumstances. Immediately after drilling into the high pressure gas area below 3750 ft. the casing pressure ranged up to 1300 lb, requiring a pull of 25,000 lb to force the drill-pipe into the hole. This meant that a pull of nearly 45,000 lb would be required to force the first few joints of 8¼-in. casing into the hole, not considering the friction of the packers. This necessitated a greater downward pull than could be exerted by the special equipment then in use at the well. To overcome this difficulty a special plug equipped with back pressure valve

was fastened to the end of the drill-pipe and run into the hole just as the drilling bit would have been run. The plug was equipped with special slips above the rubber packer which would permit it to go down into the hole, but which would not be affected by pressure exerted from below. The plug was set near the bottom of the 11¾-in. casing after the slips were set, the top portion rotated to pull a tapered mandrel upwards, thus forcing the rubber sleeves against the walls of the casing tight enough to prevent fluid or gas from coming into the hole. When the rubbers were set the drill-pipe was rotated to unscrew it from the plug and the pipe was withdrawn.

The casing was then run in, and when it met the plug it pushed it ahead and the plug acted as a casing shoe guide while shutting off all hole pressure.

In making up the string of casing an ordinary washover shoe was placed on the bottom joint, one screw type packer 21 ft. off bottom, and a back pressure valve below this packer and a special cement sleeve immediately above it. Another back pressure valve was placed 110 ft. off bottom and about 36 ft. from the top packer.

The 8¼-in. casing was run into the hole to the top of the plug and the stuffing boxes were closed. Enough pressure was applied at the top of the hole to equalise the pressure below and above the casing plug. The rubbers in the plug were expanded to the inside diameter of the 11¾-in. casing and naturally caused friction whilst going into the smaller hole. This necessitated the use of the snubbing device to apply the proper downward pressure to the casing. The casing was rotated and the two wall packers set against the formation. After cementing this string, deeper drilling was possible with a minimum effort. L. V. W. C.

1374. Extinguishing and Controlling Fire on Bell and Graddy Well. E. S. Durward. *Oil Weekly*, 1931, **63** (6), 19.

The general principles usually followed in extinguishing oil well fires are whether the fire is extinguished and the well is brought under complete control with one operation, or whether two separate operations are carried out. Only if the first method appears impossible should the second method be undertaken. The Bell and Graddy Well at Kilgore, Texas, caught fire during swabbing and cleaning operations, presumably by inflammable gas reaching the boilers and flashing back to the well.

The 7-in. casing was equipped with a casing head with two side outlets fitted with valves, both of which were open, one leading to the tanks and the other having no connexions. Through the top of the casing head 2½-in. tubing projected with a tubing gate and Christmas tree above it. The oil flow was estimated at 16,000 brls./day, with a gas flow of 6,400,000 cub. ft.

Two days after the well caught fire an unsuccessful attempt was made to extinguish the fire with 20 quarts of nitroglycerine, but the 2½-in. fittings were blown off, leaving the damaged tubing gate from which flames shot up at an angle. The gates on the side outlet of the tubing were also damaged and the flow line blown off.

The tubing gate was successfully shot off by machine gun fire allowing the flame in the tubing to shoot straight up into the air, and horizontally from the two opposite side outlets on the tubing head.

After consideration it was decided to use the tunnelling method. Considerable natural protection permitted a close approach to the fire, and a sheet-metal screen was erected above the point where the entrance for the tunnel was located. The tunnel, 6 ft. high by 4 ft. wide, was begun at a point 175 ft. from the well head, and was dug at a slight angle so that it reached the casing 25 ft. below the surface.

An electrically driven blower was installed at the mouth of the tunnel, and a pneumatic pavement drill with digging spade was used to speed up the work, and the seepage water encountered was removed by a small steam pump.

When the casing was reached, digging was continued until half the diameter of the 10-in. surface string was exposed. This pipe was surrounded with 3 ins. or 4 ins. of packed sand, etc., which had settled down around it. The mouth of the 10-in. casing was open at the surface with the 7-in. casing supported on it by a set of clamps and holding tension in the 7-in. The condition of the space between the 7-in. and 10-in. was unknown, and the possibility of hot oil passing down the annular space into the tunnel had to be considered.

A 6-in. by 8-in. nipple was first welded to the 10-in. casing, and a gate valve installed having a nipple and flange on its outer end. To this was bolted a special "Y" branch having flanges on the branch, and to this was bolted the drilling assembly. This assembly had a stuffing box and gland through which projected a spindle having a ¾-in. twist drill, upon which was threaded a washer at such a distance that the drill would pass completely through the 10-in. but not the 7-in. casing. After drilling through the 10-in. the drill was withdrawn beyond the gate. The gate was then closed and the drilling assembly removed. The annular space was then cleared by opening the gate slowly and allowing the hot water to run out.

The ¾-in. drill was then replaced by a hollow milling tool, and the drilling equipment bolted in place on the "Y" fitting. The mill carried a blind pilot in the centre to fit into the ¾-in. pilot hole already drilled. A 5·7/16-in. disc was then cut out of the casing and withdrawn with the mill.

A 6-in. line led from the "Y" branch out through the tunnel, and was connected to the flow tanks of an offset well. In order to prevent fluid of any kind coming down from above, or up from below, cement was pumped in between the 10-in. and 7-in. casings.

The 6-in. nipple welded to the 10-in. casing was cut away with sufficient of the casing to allow inserting and welding 6-in. nipples on the 7-in. casing. The cement was then chipped away and the nipple welded on. A second nipple was welded on to the 7-in about 2 ft. above the first one, and both were tested with hydraulic pressure to 700 lb/sq. in. Valves and drilling connections were then installed in the same fashion as when drilling the 10-in. casing, and similar holes were drilled in the 7-in. casing. To the lower of the two connections the same pipe connections as were used on the 10-in. string were connected, and to the upper a similar apparatus with the exception that the spindle was square. On one end of the square spindle was threaded a large nut, and on the other was a cylindrical ram similar to a blow-out preventer ram having a nose curved to fit snugly against the interior of the 7-in. casing and with a vertical groove to take 2½-in. tubing.

The ram was pushed forward by the nut on the outer end. Through the lower connection cement sacks, etc., were "lubricated," followed by water. This had no effect, as the material passed the baffle in the upper connection, due, no doubt, to the high velocity of the fluid. The baffle was then removed and the sides milled and sharp edges cut in the periphery to catch the sacks, etc. On replacing the baffle, and "lubricating" with sacks, followed by water, an immediate shut-off was effected. Pumping was continued, and in a short time the water reached the bottom of the tubing and, ascending with the flow, suddenly extinguished the fire. The casing head was then replaced. L. V. W. C.

1375. Correlation of Certain Soil Characteristics with Pipe-line Corrosion. I. A. Denison. *Bur. Stand. J. Res.*, 1931, **7**, 631–642.

An investigation has been carried out into the relationship between corrosion experienced on a group of pipe-lines in Ohio and the nature of the soils occurring in the areas under observation.

Different soils have been found to correspond in a general way to corrosive and non-corrosive areas, as shown by pipe-line replacements over a number of years, the decisive factor being apparently the acidity of the soils. The affected areas may be more or less defined by the total acidity of the soils or equally well by the p.H. values of the soil extracts in normal potassium chloride solution, although texture and drainage exert a great influence.

An accelerated test of the relative activity of soils based on the corrosion of a steel disc in contact with moist soil is described, the results of this test agreeing in general with acidity determinations in defining corrosive areas.

D. L. S.

Crude Oil.

1376. All Argentine Crudes Favour Cracking. E. F. Nelson and G. Egloff. *Oil and Gas J.*, 1931, **30** (23), 22.

Argentine crude oil was first discovered in commercial quantities in the Comodoro Rivadavia field in 1907, when Government employees looking for fresh water struck a strong flow of heavy oil at 1739 ft. As a result 12,000 acres of the surrounding territory were reserved for Government use. In 1914–15 the Cacheuta field in the province of Mendoza was brought in, followed by the Plaza Huincul field in Neuquen in 1918. In the northern provinces of Salta and Jujuy commercial production was not obtained until 1926. Seams of Rafaelite, a paraffin base asphaltite, similar to Grahamite, are found at Auca Mahuida.

Comodoro crude is of the nature of a heavy fuel and possesses the following properties (ref. B. of Mines Tech. Paper 346): A.P.I. gravity, 20·8; pour point, 15° F.; water, 0·02%; visc. Saybolt at 100° F., 2490 secs.; sulphur, 0·17%; carbon residue, 10·5%. The following products may be obtained from it: light gasoline and naphtha (A.P.I., 55·7°), 4·9%; kerosine (A.P.I. 45·8°), 1·7%; gas oil (A.P.I. 34·6°), 0·9%; lubricating oil dist., 19·2%; and residuum (A.P.I. 15·4°), 63·2%.

Cracking tests by the non-residuum method on a mixture of 93% fuel oil and 7% gas oil gave a yield of 76·2% of pressure distillate at 200 lb. pressure and 895° F.. Coke, gas and loss amounted to 23·8%. On distillation of the treated pressure distillate a yield of 57·9% of gasoline (based on intake) was obtained. The gasoline had a gravity of 60·0 A.P.I. and boiling range 89° F.–437° F. The pressure distillate is treated with 3–5 lb. 66° H_2SO_4 per brl., followed by 1·5% of 16° Bé sodium plumbite solution, before distillation. The freshly distilled gasoline should be washed with 1% of 1–2° Bé caustic soda solution to improve the odour.

Plaza Huincul crude oil is lighter than Comorodo oil and has the following properties: A.P.I. gravity, 33·8; pour point, below 5° F.; visc. Saybolt U. at 70° F., 75 secs. and at 100° F., 55 secs.; sulphur, 0·29%; water, nil; carbon residue, 9·9%. The following products may be obtained from it: light gasoline (to 212° F.) (A.P.I. 70·6°), 5·2%; gasoline and naphtha (A.P.I. 56·7°), 25·1%; kerosine (A.P.I. 43·6°), 10·1%; gas oil (A.P.I. 37·4°), 16·6%; lubricating oil distillate, 16·6%.

Mendoza crude possesses the following properties: A.P.I. gravity, 30·8°; pour point, 55° F.; water, nil; visc. Say. Univ. at 70° F. at 33 secs. and at 100° F., 71 secs.; sulphur, 0·19%; carbon residue, 8·6%. On distillation the following products are obtained: gasoline and naphtha (A.P.I. 53·2°), 17·5%; kerosine (A.P.I. 44·5°), 11·9%; gas oil (A.P.I. 36·8°), 15·8%; lubricating oil distillate, 23·1%; residuum, 32·2%.

Salta-Jujuy crude (see *Journ. Inst. Petr. Tech.*, 1924, **10**, 829) has A.P.I. gravity, 15·9°; visc. Engler at 50° C., 350·6°; calorific value, 10,719 cals./gr.; sulphur, 0·527%; water, 4·90%.

On fractional distillation 3.40% distils to 347° F., 12% at 347° F.–572° F., and 17% at 572°–680° F., and residuum 62·7%.

The total production of crude oil in Argentina during 1930 is estimated at 9,400,000 brls., while there are fifteen refineries, three of which are under Government control. The largest is the La Plata refinery of the Yacimentos Petroliferos Fiscales (Argentine Government) with a capacity of 14,000 brls. daily. C. L. G.

1377. Separation of Oil. A. B. Jones and Industrial Association. U.S.P. 1,824,780, 29.9.31. Appl., 24.2.28.

A process is described for the quick and economic separation of the various fractions of crude petroleum without injuring or cracking the oils. The liquid having as one fraction the desired oil, is brought into contact in a highly dispersed form in a treating chamber with a gaseous medium previously heated to a temperature sufficiently high to vaporise the desired oil and thus separate it from the remaining liquid. The distillate thus obtained together with the gaseous medium are passed to a fractionating column and from thence to a cooling zone into which liquid of a lower boiling point is sprayed. W. D. S.

Gas.

1378. Girdler Process in Solidified Carbon Dioxide Plant. F. R. Staley. *Petr. Eng.*, 1931, **2** (13), 20.

The Carbonic Products Co. recently commenced operating its plant at Tulsa, for the manufacture of solid and liquid CO_2 from natural gas free from sulphur. The plant treats 175,000 cu. ft. of gas and produces 6 tons of solid and liquid CO_2 per day. The gas is burned under a boiler which supplies power for the plant, and the flue gases, which contain about 10% of CO_2 and 89% of N_2, are drawn from the stack through a scrubber by means of a Roots blower. They are then discharged through a second scrubber to a bubble type absorption tower, the scrubbed gases entering at the bottom, whilst an aqueous solution of one of the ethanolamines is pumped over the top tray. The CO_2 is absorbed by the chemical and the solution pumped to the reactivator tower through heat exchangers against reactivated chemical. The reactivator tower is also of bubble type and is heated by means of steam coils situated at the bottom. Pure CO_2 leaves the top of the tower under a pressure of 2 lb. and passes to compressors where it is liquefied under a pressure of 1100 lb./sq. in. The Frick process is employed to produce solid CO_2 from the liquid product. The CO_2 is absorbed by the Girdler process which utilises the method of extraction by means of triethanolamine or diethanolamine. The physical properties of the ethanolamines are given. In plant operation for extraction of CO_2 a 50% solution with water is generally used. It has been found experimentally

that triethanolamine will absorb CO_2 in the proportion calculated theoretically, viz., 1:90 by vol., but diethanolamine will absorb 140 vols. of CO_2 at ordinary temperatures, which is slightly higher than the quantity calculated theoretically. The presence of water is necessary in order to obtain any reaction, and the amount of CO_2 held in combination by water solution of the ethanolamines will vary with temperature and concentration of CO_2 in the gas mixture. Reactivation is easily and completely accomplished by suitable plant with negligible decomposition, losses may occur by leakage or carrying over solution from the absorber reactivator, and the Girdler Corporation estimates a liberal allowance for loss of about 50% per annum of the original solution charged. Details of the plant and of the methods of handling the solid and liquid CO_2 are given, together with the properties of, and an outline of the commercial applications of, the solid material.

R. A. E.

1379. East Texas Pools have Peculiar Gas. L. G. E. Bignell. *Oil and Gas J.*, 1931, **30** (19), 30.

The gas from the East Texas field is characterised by low methane content, the analyses averaging only about 43% methane, 13% ethane, 22% propane, 14% butane and 8% pentane and heavier products. It has been determined that the original pressure in this field is of the order 1560/1600 lb., while the temperatures at the bottom of the wells have been found as high as 162° F. At this temperature and pressure all gases heavier than ethane would be liquefied and would exist in that form along with the crude oil. The resulting reduction in the viscosity and specific gravity of the crude would enable it to retain a higher percentage of methane and ethane than that shown above. Work on the solubility of gas in oil under East Texas field conditions has shown that the natural crude oil is not saturated with gas, so that there can be no free gas existing with the oil in the sands of the East Texas field.

The low methane content of the gases is attributed to migration of the light ends of the gas through the east end of the pool to the top of the Sabine uplift where there are now very prolific gas fields containing 97% methane. The fact that only the light fractions escaped may be attributed to the presence of fine volcanic ash which filled the pores of the East Texas sand during earlier geological deposition. Alternatively the oil as formed may have been of low methane content, owing to the escape of marsh gas to the atmosphere when the oil was formed.

C. L. G.

1380. Mixing Natural and Coal Gas to Supply the City of Chicago. H. E. Bates. *Oil and Gas J.*, 1931, **30** (20), 17.

Chicago is supplied with a 45%/55% mixture of coal gas with heating value of 550 B.Th.U.'s and natural gas with heating value 1000 B.Th.U.'s. The natural gas is pumped from the Texas Panhandle field and arrives via a 24-in. line at the Calumet Station of the Peoples Gas Light & Coke Co., another line going to the Crawford Avenue Station of this company on the other side of Chicago. The Calumet Station receives 70,000,000 cu. ft. of gas daily purchased from local gas concerns, while the Crawford Station has a coal gas plant of 24,000,000 cu. ft. and a water gas plant of 40,000,000 cu. ft. daily capacity. Normally only the coal gas is mixed with the natural gas, but, during peak load requirements, water gas is also used.

Mixing of the natural and manufactured gas is effected by a Smoot gas-mixing control, a constant heat value in the mixed gas being maintained by a balanced valve regulator controlled either manually or automatically from a calorimeter.

The gas before delivery into the transmission system is treated with an oil fog to prevent the drying out of the yarn packing in bell and spigot joints. The oil fog is obtained by bubbling a portion of the gas through hot oil at 450/500° F. and mixing it with the remainder of the cold gas, whereupon the oil vapour condenses, the heavier particles of fog being then removed by a cyclonic separator. The gas is compressed to 20 lb. or 25 lb. to the square inch, and passes to Griscom Russell coolers, where the gas is cooled from 240° F. to 100° F. C. L. G.

1381. Manufacture and Production of Liquid, in Particular Aromatic Hydrocarbons. I. G. Farbenind. A.-G. E.P. 357,170, 10.9.31. Appl., 10.6.30.

Liquid, in particular aromatic hydrocarbons, are obtained by the continuous thermal treatment of gaseous hydrocarbons of the methane series or gases rich in methane, in the presence of a catalyst consisting of free elementary silicon. The catalyst may be employed in various forms, as, for example, in the form of pieces or moulded bricks obtained by fusing silicon powder with or without the addition of a binder such as clay, alumina, water glass or other silicates. The temperatures employed range between 700° C. and 1200° C., the highest yields from gases rich in methane being obtained between about 1040° C. and 1080° C. If the operation is carried out under pressure, lower temperatures may be used, the optimum temperature being determined by the pressure. S. F. B.

See also Abstract No. 1447.

Motor Fuels.

1382. Use of Alcohol as a Motor Fuel. P. Dumanois. *Rev. Petrolifere*, 1931 (444), 1323-1328, 1375.

A discussion is given of the employment of alcohol in motor fuels in France and of work on this subject carried out in the French national laboratories. It is shown from the shape of the power-mixture-strength curves that alcohol fuels are less sensitive, as regards power output, to changes of mixture-strength due to carburettor setting, speed and external conditions, than is petroleum spirit. When about 10% of alcohol is mixed with gasoline no adjustment is required to the jet, as the influences of increased density and viscosity practically cancel out. The anti-detonating value of the fuel is improved and the power is increased. Another advantage is that the tendency to form ice-crystals in the jet, in certain conditions, is greatly reduced owing to the solubility of water in alcohol. For practical use the double carburettor of Frank Lloud has been suggested, enabling moist alcohol or other fuels immiscible with gasoline to be burned simultaneously. In 1923 "Carburant National" (50/50 alcohol-gasoline) was introduced, but has been unsuccessful chiefly owing to the facts that adjustment of jet is required for a mixture containing this proportion of alcohol and that the distribution is difficult. The use of smaller proportions of alcohol would avoid these difficulties, but the mixture is then not sufficiently stable to water, *e.g.*, with 5 or 10% of 99·4% alcohol the mixture separates on addition of 0·2% water. Carburant National required about 2% of water before separation occurred. Data is given on the stabilities of various mixtures. With a mixture containing 25% of alcohol, 0·6% of water, or alternatively,

a reduction of temperature to about −26° C., is required to cause separation. With 0·35% accidentally acquired water, the mixture is still stable down to −15° C. The law of July, 1931, consequently required importers of gasoline to add 25 to 35 parts of alcohol to 100 parts of gasoline, *i.e.*, the mixture contains 20–26% of alcohol. An adjustment of the jet is necessary for this mixture. The octane number of the blend, even if a bad gasoline is employed, is about 80; practically the same volumetric consumption is obtained as with gasoline, with far less heating of the engine. If the new Carburant National be mixed with gasoline in the proportion of two to one, it is not necessary to readjust the jet. The stability of this mixture, which contains about 16% of alcohol, is poor, but in practice separation is not likely to occur.

Mixtures of synthetic methanol and benzole with gasoline are also discussed. The use of alcohol mixtures is recommended on national grounds.

E. B. E.

1383. Solubility of Water in Aviation Gasolines. E. W. Aldrich. *Ind. Eng. Chem. Anal. Edn.*, 1931, **3**, 349.

The gasoline is saturated with water at the desired temperatures and sodium-potassium alloy free from oxides is added to the sample after the removal of dissolved gases. The hydrogen evolved is collected and measured.

A. R. S.

1384. Aviation Gasoline Specification. *Oil and Gas J.*, 1931, **30** (23), 90.

The U.S. Bureau of Mines proposes to supersede the present specification for fighting grade aviation gasoline by one which includes the following points: (1) An anti-knock value of not less than 87 octane number; (2) permission to add ethyl fluid in such quantity that the concentration of L.T.E. does not exceed 6 ml. per U.S. gal.; (3) permission to add benzole, etc., in such quantity that the freezing point of the blend is not higher than −60° C.; (4) the gasoline to be tinted a shade of blue of a certain minimum intensity; (5) no grey or black corrosion on evaporation from copper dish; (6) a maximum of 10 m.g. of gum per 100 ml. of gasoline after subjection to an accelerated ageing test for 4 hrs. at 100° C. with oxygen at approx. 100 lb. pressure; (7) sulphur content, max. 0·10%; (8) max. vapour pressure Reid at 100° F. not exceeding 6·5 lb. per sq. in.; (9) minimum percentages distilling over at various temperatures in A.S.T.M. distillation are: 10% at 75° C., 50% at 100° C., 90% at 135° C. The distillation loss is added to the percentage distilling over at each temperature, and the residue must not exceed 2%. The methods to be employed in carrying out each test are described in detail. Volumetric and gravimetric methods are given for the estimation of L.T.E. The distillation method varies slightly from that given in method 100:13. The anti-knock tests are carried out in an 8.30 engine, and the temperature of the cooling medium is maintained at 150° C. This is necessary on account of the relative deterioration in anti-knock value at high cylinder temperatures of fuels owing their anti-knock property to their content of cracked or aromatic products. The test method only partly duplicates the relative deterioration observed in tests of hot-running multi-cylinder aircraft engines, and is therefore somewhat arbitrary and will be modified later to allow the use of alternative methods. The addition of L.T.E. to a gasoline containing any considerable quantity of aromatic or unsaturated compounds does not, in general, diminish the relative loss of anti-knock value. A temperature plug is used for measuring knock. The Reid vapour-pressure maximum is necessary because present fuel systems in Air Corps equipment were developed on fuel with 4–6 lb. Reid vapour pressure.

R. A. E.

1385. Colouring Gasoline or other Petroleum Distillates. A. L. Mond, Assr. of Patent Fuels and Color Corpn. E.P. 357,179, 17.9.31. Appl., 17.6.30.

It is claimed that gasoline can be coloured with organic dyestuffs which normally precipitate from such gasoline by the addition of a water insoluble soap, such as aluminium stearate, calcium oleate, etc. The dye and stabiliser are made up in benzole solution before addition to the gasoline. Specific organic dyes are mentioned, including: arylated rosanilines, basic triphenyl methanes, indulines, rhodamine, acridines, etc., and it is also stated that, if flourescence is one of the properties of the dye, the addition of the stabiliser serves to bring out this phenomenon. H. C. R.

1386. Liquid Fuel. J. L. Whiteman. E.P. 357,453, 24.9.31. Appl., 31.12.30.

Liquid fuels suitable for I.C. engines are produced by mixing crude oil or oils, such as petroleum, shale oil or oils produced by the distillation of coal, with one or more of the lower monohydric aliphatic alcohols in such proportions that the tar and other normally insoluble constituents of the crude oil, *e.g.*, wax, are dissolved or held in suspension thereby. S. F. B.

1387. Diminishing the Explosiveness of Explosive Gaseous Mixtures. W. P. Jorissen and J. Booy. E.P. 357,530, 14.9.31. Appl., 14.5.30.

The tendency of certain gaseous mixtures which may or may not contain finely divided particles or liquid drops in suspension to explode may be diminished or entirely prevented by the addition of a small quantity of one or more halogen or phenolic derivatives or both to the mixture.

S. F. B.

See also Abstract No. 1450.

Kerosine.

1388. Catalytic Decomposition of Kerosine. E. W. Kanning and O. W. Brown. *J. Physical Chem.*, 1931, **35**, 2689–2710.

This research was undertaken to determine the most suitable temperature for the decomposition of a kerosine fraction boiling between 200 and 230° C. when passed over cobalt, iron, manganese or nickel catalysts. The effect of change of temperature on the volume and composition of gaseous products was studied. The volume of gaseous product obtained from 10 c.c. of the kerosine, %H, $\%CH_4$, saturated gaseous hydrocarbons above methane in the paraffin series and unsaturated hydrocarbons in the gaseous product, the volume of liquid product and the density and degree of unsaturation of the liquid product as shown by the iodine number were determined. In each instance the weight of carbon deposited on the catalyst from a given volume of kerosine was ascertained.

With all four catalysts it was found that an increase in temperature gave an increase in the quantity of kerosine decomposed. Carbon, when deposited at low temperatures, rendered the catalyst less active, while that deposited at higher temperatures had little or no effect. The four catalysts can be arranged as follows as to their ability to decompose the kerosine fraction referred to: cobalt, iron, nickel and manganese.

Cobalt was shown to be the best catalyst, since it gave the greatest volume of gas in the shortest time. The conversion was continuous at 500° C., 1 g. of cobalt catalyst converting 0·33 g. of kerosine into 6·3 c.c. of gas in one minute.

Only nickel gave unsaturated hydrocarbons in the gaseous product to any extent; cobalt gave traces. The gas having the highest percentage (46·4) of methane was obtained over nickel at 390° C. The gas having the highest percentage of hydrogen, 90·5, was obtained over iron at 400° C. Nickel and manganese at 550° C. favoured the production of large quantities of paraffin hydrocarbon gases above methane. W. S. E. C.

Lubricants and Wax.

1389. Lubricants and Cylinder Stocks Recovered under High Vacuum. J. W. Beall. *Refiner*, 1931, **10** (10), 72.

Certain problems attendant to vacuum distillation procedure are dealt with, although no attempt is made to give a complete and comprehensive treatise on the subject. Initially certain fundamentals of the subject of preparation of lubricants by means of vacuum distillation are discussed. Practical suggestions for the design, construction and operation of "wet" and "dry" vacuum units follow, while such practices as flow of oil through the unit, reboiling, refluxing, heat exchange, the use of steam or such heating media as diphenyl and mercury, are critically discussed. Vapour pressure curves for various conditions are given and a typical diagrammatic flow chart of a "dry" vacuum unit designed for the preparation of lubricants by vacuum distillation is also presented. W. D. S.

1390. Vacuum Distillation: Its Effect upon Lubricating Stocks. M. B. Muller. *Refiner*, 1931, **10** (10), 95–97.

The idea of producing heavy lubricating stocks by vacuum distillation is fostered by the desire of concentrating asphaltic impurities in a still residue so that the overhead stocks can be brought to final colour with less loss and less expense than when residues of similar character are treated. Thus vacuum distillation for the production of heavy overhead lubricating oils is limited to the handling of mixed or asphalt-base crudes wherein, if similar oils were produced as a residuum the loss from subsequent acid treatment would be far in excess of that experienced where the overhead stock is produced instead. Acid treatment is cut down considerably and loss to sludge is minimised. Doubt has been expressed as to whether the resulting product could be successfully centrifugally dewaxed, but it is shown that by improvements in the design of the vacuum still this difficulty can be largely overcome. The problem is not entirely solved, but it is suggested that the final solution will come when it has been commercially established that it is feasible to produce from a crude a base overhead lubricating stock which can after dewaxing be subsequently fractionated by further vacuum distillation into each of the various grades of lubricating oil required. W. D. S.

1391. Oil Consumption as Affected by Engine Characteristics. H. C. Mougey. *J.S.A.E.*, 1931, **28**, 200–214.

The author believes that the oil consumption problem has to be solved before the winter-starting and winter-lubrication problems, since the two last named require light oils which have high consumptions. The principal factors involved in oil consumption in the order of their importance are: (1) Engine speed—other things equal, an increase of speed from 30 to 55

m.p.h. can increase the oil consumption approximately seven times. Important is the finding that consumption tests on engines run on the bench do not have any close relation to the consumption on the road, since the windage past the crankcase openings in the latter conditions increase the oil consumption; (2) Oil leaks; (3) Design of the lubricating system and changes due to wear: full splash feed systems tend to be accompanied by lower consumption than pressure feed systems; (4) Viscosity of the oil: high viscosity decreases the quantity of oil thrown on the cylinder walls and that lost by leakage, but it also increases the difficulty of removing or draining the oil from the cylinder walls. Where these two opposed factors cross, indicates the maximum gain in the reduction of oil consumption through increased viscosity. Further increase of viscosity beyond this point increases the consumption. The author further deals at length with the question of viscosity-temperature characteristics, and the temperature range between the lower limit, at which the oil has the maximum viscosity of 50,000 sec. Saybolt, beyond which the minimum cranking speed for starting is difficult to obtain, and the higher limit beyond which the oil viscosity is too small to allow safe lubrication; (5) Volatility: differences in volatility between oils has little or no effect if the crankcase oil temperature is below 170° F. Any tendency towards higher sump temperatures can be obviated by oil coolers; (6) Pour point: affects consumption indirectly—*i.e.*, via its effect on engine wear; (7) Possibility of effects from carbonisation of oil control rings: any clogging of holes and slots and sticking of the oil rings in the grooves decreases the effectiveness of the rings and increases oil consumption. Better cooling of piston and rings is believed to decrease the formation of carbon in the grooves. Increasing the flow of oil through these grooves, clearances, etc., would tend to flush away any carbon tending to accumulate; (8) Length of time between oil changes: the author believes that with engines which run under normal conditions of speed, etc., leading to heavy oil consumptions, any ordinary frequency of oil changing would have little effect on the quantity of oil consumed. But draining of the crankcase in the winter season may have serious effects: (*a*) the new oil, having a higher viscosity than the diluted rejected oil, might increase starting difficulties; (*b*) the higher viscosity of the new oil would tend to increase the lag between the starting of the engine and the establishing of adequate supply of oil to the bearings. The more extended is the lag, the greater are the possibilities for excessive bearing wear.

C. I. K.

1392. Manufacture of Lubricating Oils. Standard Oil Co. (Ind.). E.P. 355,214. Appl., 18.2.30. Aug. 18, 1931.

Hydrocarbons containing not less than 30 per cent. hydrocarbon wax are cracked to unsaturated hydrocarbons and then polymerised in the presence of a polymerising agent—*e.g.*, $AlCl_3$. The polymerisation is carried out at atmospheric pressure or at pressures preferably below 100 lb./sq. in., and the temperature employed varies between 750 and 1100° F.

W. D. S.

1393. Refining of Wax-containing Oil. Sharples Speciality Co. E.P. 356,172, 28.8.31. Appl., 28.4.30.

Low cold test oil is produced from wax-containing mineral oil by a continuous distillation method. The oil is heated in a tubular heater to avoid cracking, and the resulting liquid and vapour are fractionated by passing into a fractionating tower with reflux. Adjacent fractions are produced containing respectively wax that is removable by amorphous wax-removing methods and wax that is removed by crystalline wax-removing methods.

W. D. S.

1394. Recovery of Paraffin Wax. J. Y. Johnson and I. G. Farbenind. A.-G. E.P. 356,205, 4.9.31. Appl., 4.6.30.

Material containing substantial amounts of paraffin wax and other carbonaceous substances is mixed with a mixture of organic solvents. This mixture consists of the formates of lower alcohols of the fatty series and other solvents having a greater solvent power for asphaltic bodies than the formates. Examples of these latter bodies are benzine, benzene, carbon disulphide or carbon tetrachloride, and they need only be added in small amounts to the mixture; 2-20 per cent. reckoned on the amount of purifying mixture employed is sufficient to improve the quality of the paraffin wax. W. D. S.

1395. Lubricating Oils. Standard Oil Dev. Co. E.P. 357,717, 1.10.31. Appl., 9.9.30.

A modification of a method for hydrogenation of lubricating oils as claimed in E.P. 349,342 (*Abs.* 982) is described. Tables are given showing the qualities which may be expected by the improved process. W. D. S.

1396. Manufacture of Lubricating Oils. E. M. Clark and Standard Oil Dev. Co. U.S.P. 1,820,963, 1.9.31. Appl., 22.1.27.

A lubricating oil distillation apparatus is described. The oil is heated rapidly to its vaporising temperature and passed into a fractionating column at a pressure of 25 m.m. Hg. Steam is injected into the lower portion of the column. The lubricating oil fractions are condensed and treated while still hot with an alkaline solution, subsequently distilling at an absolute or partial pressure of 15 m.m. Hg. W. D. S.

1397. Purification of Oil. L. H. Clark and Sharples Speciality Co. U.S.P. 1,823,087, 15.9.31. Appl., 4.2.26.

An apparatus is described for the purification of oil used for the lubrication of cylinders of light hydrocarbon internal combustion engines. The oil is first washed with an aqueous alkaline reagent capable of rendering immiscible impurities separable from the oil and then pumped to a centrifuge for separating the impurities and the reagent from the oil. The soluble contaminating substances are removed by distillation. The oil discharging from the centrifuge is charged at once to the still by valve controlled pipes. W. D. S.

1398. Purifying Crank Case Oil. D. S. Belden and Filtrol Co. U.S.P. 1,823,185, 15.9.31. Appl., 8.6.29.

The crank case oil is mixed with about 5% by wt. of an acid-activated clay and heated to about 600° F. while a lively stream of inert permanent gas is bubbled through. The lighter fractions are thus distilled off and the purified oil may be filtered from the clay. W. D. S.

1399. Treating Naphthenic Oils. N. E. Lemmon and Standard Oil Co. (Ind.). U.S.P. 1,823,614, 15.9.31. Appl., 12.5.28.

Lubricating oils with a substantial degree of naphthenic acidity are given a light sulphuric acid treatment (0·25 lb. of 89% H_2SO_4 per gal. of oil), the sludge removed by settling and the oil neutralised with caustic soda solution. The naphthenic acid compounds are then removed from the oils by treatment with aqueous solutions of alkali salts of preferentially water soluble sulphonic acids. The sour oil is then further treated with 0·1 lb. H_2SO_4 (89%) per gal. of oil, settled to remove sludge, and contacted with 10 lb. of clay per 100 lb. of oil at about 150° F. W. D. S.

1400. Removal of Naphthenic Acids from Hydrocarbon Oils. N. E. Lemmon, F. V. Grimm and Standard Oil Co. (Ind.). U.S.P. 1,823,615, 15.9.31. Appl., 14.5.28.

Naphthenic acids (acidity 0·2 mg. KOH per g. of oil) are removed from hydrocarbon oils by a mild H_2SO_4 treatment (0·5 lb. H_2SO_4 of 80–90% strength per gal. of oil). The naphthenic acids are thus converted to a form readily removable by contact clay. Care must be taken to avoid acid treatment of such severity that the clay is rendered ineffective in the removal of naphthenic acids. W. D. S.

1401. Treating Hydrocarbon Oil. W. M. Stratford and Texas Co. U.S.P. 1,823,994, 22.9.31. Appl., 13.1.28.

A process for purifying lubricating oils is described. The preliminary treatment depends upon the stock, but it may be necessary to treat the oil with sulphuric acid in the usual way. The essential part of the process consists of mixing the oil with an adsorbent decolorising material which is most efficient at temperatures in excess of about 250° F. The mixture is heated to 500° F. or above and then subjected to distillation under a vacuum, in the presence of an alkali, without substantial decomposition. W. D. S.

1402. Treating Hydrocarbon Oils. R. E. Manley. U.S.P. 1,825,324, 29.9.31. Appl., 16.11.27.

In dewaxing the more viscous residual and distillate oils which have not been subjected to an appreciable amount of cracking it is found that the ordinary methods of chilling and filtering are impracticable. Wax found in these products is usually referred to as amorphous wax. It has been found that wax of this nature can be filtered and dewaxed in the presence of a diatomaceous earth. The process consists in mixing the cylinder stock or other raw material with naphtha. The diluted oil is forced through a chiller and cooled to the degree necessary to yield a product of the desired setting point. From the chiller the oil passes to a tank wherein the earth, to the extent of 10 to 30 lbs./brl. of diluted stock, is mixed. The mixture is filter pressed, forming a porous cake of wax and comminuted solid material. When the proper thickness of cake has been built up on the filter the oil supply is cut off and the filter drained. Cold compressed air or other gas is turned into the filter to displace the oil remaining in the filter. Thereafter cold naphtha is pumped through the filter to dissolve any oil left in the cake.

The cake may then be blown with cold air to displace excess naphtha, providing a dry filter cake of wax and diatomaceous earth which is easily removed.

The earth may be recovered and returned for further use. R. P.

1403. Production of High-Melting Paraffin and Ceresine. A. Loebel and N.V. De Bataafsche Petr. Mij. U.S.P. 1,825,533, 29.9.31. Appl., 22.10.28.

From a paraffinic crude oil the benzine, kerosine, gas oil and paraffin oil are distilled off (those fractions boiling up to 400° C. approx.), and the residue of mixed base oil treated with Edeleanu-extract, boiling above 200° C. (hydrocarbons, b.p. $>200°$ C., which are materially soluble in liquid SO_2). The mixture is subsequently distilled and the hard paraffin may be separated from the distillate. The residue left in the still is a hard asphalt. W. D. S.

1404. Extraction Process. R. de M. Taveau and Texas Co. U.S.P. 1,825,762, 6.10.31. Appl., 15.5.25.

An apparatus and process is described for the recovery of valuable lubricating oil from an asphalt base residuum. The latter is treated with isopropyl alcohol and the oil-alcohol solution separated from the undissolved asphaltic matter. The isopropyl alcohol is evaporated from the purified oil, and by using a closed system the losses are infinitesimal. W. D. S.

See also Abstract No. 1445.

Fuel Oil.

1405. Apparatus for Generating and Burning Liquid Fuel. H. Mayer. E.P. 354,359. 6.8.31. Appl., 6.5.30.

Liquid fuel is vaporised by being dripped on to a hot plate having air passages through it. This device is placed in a chamber in which a rich mixture of vapour and air is formed. In this invention, a vapour guide open at one side for directing the rich mixture into a mixing chamber, is placed over the vaporising plate. Round the vapour guide is situated a channel for supplementary air which is also open to the mixing chamber. Baffles are fixed in the channel so as to restrict the outlet to the mixing chamber. W. D. S.

Fluxes and Asphalts.

1406. Asphalt, Bitumen and Tar, Their Reciprocal Value in Bituminous Road Construction. A. W. Attwool and D. C. Broome. *Roads and Road Constr.*, 1931, **9**, 303–305, 339–340.

Many of the handicaps of tar as "road binders" are removed by the addition of suitable proportions of asphalt or bitumen. The tars so treated are less susceptible to temperature changes, the adhesive properties are improved and the resistance to oxidation and weathering is increased. Tables are given which show the wide variation in the physical characteristics of typical tars and bitumens employed in modern practice.

Viscosity is the most suitable criterion of the suitability of a tar for any class of work, while for road work it should be as high as is consistent with ease of handling, etc. The colloidal suspension of free carbon is an essential constituent of a good road tar, Vertical retort tars need reinforcing in this respect by the addition of certain native asphalts which themselves contain free carbon, insoluble organic material, and colloidal mineral matter. Residual bitumen is less effective than native asphalts.

The effect of the addition of a typical tar to different types of bitumen is illustrated. Native asphalts combine thoroughly with most tars, but residual bitumens frequently coagulate, forming "islands" which absorb free carbon and leave a medium of tar oils entirely devoid of free carbon. This is illustrated by photomicrographs of mixtures of tar and bitumen.

The most suitable types of dressing are described together with their methods of application.

Consideration must be given to the length of time the mixtures will be in storage or transit and the viscosity adjusted accordingly. Under similar conditions viscosity should be slightly higher for a granite aggregate than for limestone, and lowest for slag. For mixtures designed along the lines of hot asphalt specifications, the methods of application are similar to those employed for normal tarmacadam mixtures apart from the temperature modification. A comparison of the composition of tar mixtures and hot asphalt preparations for base coat and single-coat pavement, is shown. The present system of testing tar-bitumen mixtures from the standpoint of existing tar or bitumen practice is inadequate in gauging their true characteristics. According to present methods, the viscosity is determined at one arbitrary temperature only; this gives no indication of the susceptibility to temperature changes.

The viscosity and other characteristics should be determined over the widest temperature range. This is difficult with the forms of viscometer in use, and it is interesting to note that a new instrument has been developed in the laboratories of the Limmer & Trinidad Lake Asphalt Co., Ltd., in which the absolute viscosity of tars, oils, bitumens, etc., can be determined by means of a falling sphere, the fall being "observed" electrically. Subject to certain conditions any size ball may be used and the tests carried out at any temperature, the viscosity at any other temperature being calculable from the results obtained over the convenient temperature range.

A photograph of the apparatus is shown and details will be published in the near future. A. O.

1407. Bituminous Concrete. E. R. Taylor. E.P. 357,282. 24.9.31. Appl., 1.7.30.

A process is described for mixing bituminous concrete wherein is incorporated a quantity of calcined material to adequately absorb the moisture in the mineral aggregate. Subsequently the requisite volume of cut-back bitumen is added. H. C. R.

Special Products.

1408. Synthetic Rubber from Petroleum. C. K. Francis. *Oil & Gas J.*, 1931, 30 (19), 24.

The world now requires over 857,000 tons of rubber per annum. The major proportion is used in the U.S. which produces only 3% of the supply. The sources of the natural product and the average market prices for the last twenty years are given. It appears impossible to produce a synthetic material at a price competitive with that of the natural product, but the possibility of synthetic production would be of strategic importance. A consideration of the carbon/hydrogen ratio of natural rubber (87.91/11.86) suggests a hydrocarbon of general formula $(C_{10}H_{14})x$, and indicates that a commencement in the synthesis could be made with the aid of a hydrocarbon having the formula $C_{10}H_{16}$. The arrangement of the fundamental molecule of rubber suggested by Harries is illustrated. It is known that methylbutadiene after long standing will change to rubber. The theory of the formation of rubber in the plant is discussed: it is assumed that butadienes are first formed and then polymerised with the aid of a catalyst and the action of sunlight. From pentane, which can readily be obtained from petroleum, it appears possible to obtain the group C_5H_8, which appears in the fundamental rubber molecule of Harries, by treatment with acid according to the principles

established for sugar synthesis, but the separation from petroleum of immense quantities of pentane required to build a complex molecule having a mol. wt. equal to that of rubber (*i.e.*, about 6,000) is obviously difficult. Synthetic rubber products so far prepared lack the elasticity of the natural material possibly owing to the absence of resins and nitrogenous substances which are present in small percentages in the natural rubber. Acetone which is indirectly obtainable from petroleum has been the starting point of many syntheses; reduction to dimethybutadiene being first effected and followed by polymerisation.

When cracking petroleum fractions small percentages of diolefines, butadiene, methyl- and dimethyl-butadiene can be obtained. When these compounds are allowed to remain in contact with metallic sodium for several months a material which is suitable for use as a basis in the manufacture of hard rubber articles is obtained. Reference is made to two patents which have been granted for the manufacture of rubber substitutes from isoprene and turpentine respectively.

The basic materials required in the production of synthetic rubber are thus all obtainable from petroleum, but in comparatively small quantities and at high cost. The plant required for conversion is very expensive and long treating periods are necessary, so that commercial production does not appear feasible. R. A. E.

1409. Manufacture and Production of Carbon Black. J. Y. Johnson. E.P. 358,178, 8.10.31. Appl., 28.7.30.

Carbon black is produced by passing the gaseous hydrocarbon to be decomposed counter current to a stream of catalyst, consisting of such metallic oxides as iron, nickel, cobalt and zinc. The catalyst chamber is held at approximately 380° C. and the catalyst is reduced by hydrogen at the start of the operation and by hydrogen, produced in the decomposition when the plant is operating. The carbon black is delivered along with a quantity of catalyst. Mechanical and wind sifting is used to separate catalyst and product. A. R. S.

Refining and Refinery Plant.

1410. Manufacture of Hydrocarbons of Higher Molecular Weight from Unsaturated Hydrocarbons. N. V. de Bataafsche Petr. Mij. E.P. 358,086, 29.9.31. Appl., 29.4.31.

A process for the polymerisation of hydrocarbons, in particular olefines, by treating them in the presence of catalysts at normal or increased pressures, is described. Metal halides (especially those of metal of valency 3 or more) are used as catalysts, and in order to moderate their polymerising action, complexes with polar compounds are also used. The latter should not combine with the unsaturated hydrocarbons under the conditions of reaction. Examples of the polar organic compounds are nitrobenzene, nitromethane, acetone, benzophenone, diphenylsulphone, etc. W. D. S.

1411. Carrying out Distillations under Low Pressure. N. V. de Bataafsche. Petr. Mij. E.P. 358,391, 8.10.31. Appl., 22.1.31.

A process and apparatus is described for distilling under low pressures in which the velocity with which the transport from the vaporising to the condensing surface takes place is substantially equal to the molecular velocity. This is achieved by making provision to avoid the vaporisation being retarded by diffusion in the distillate, for instance, by providing for

sufficient turbulence of the distillate or by working with thin distillate layers, the rate of distillation then being substantially equal to the molecular velocity. Vaporising and condensing surface are arranged in an evacuable chamber, such that the average distance which the molecules of the vaporised material have to travel between those surfaces in the direction in which the vacuum pump is working, is always greater than that in the opposite direction; both distances are of the absolute order of the mean free path of the molecules under the conditions prevailing. W. D. S.

1412. Vacuum Distillation of Oils. A. E. Harnsberger and Pure Oil Co. U.S.P. 1,822,733, 8.9.31. Appl., 20.10.27.

An apparatus for the distillation of mineral oils under vacuum is described. It consists of a pipe still formed of a plurality of groups of pipes and means of serially connecting the several groups of pipes. The number of pipes in the several groups successively increases along the length of the still and the diameter of all the pipes is kept constant. Such a degree of vacuum is maintained at the discharge ends of the pipes as will penetrate back to the first pipe. The vacuum is in such a degree that vaporisation takes place at a temperature of 750° F. W. D. S.

1413. Apparatus for Contacting Vapours with Solids. T. T. Gray, Assr. to Gray Process Corpn. U.S.P. 1,823,895, 22.9.31. Appl., 28.8.26.

An apparatus is described for contacting hydrocarbon vapours with a powdered treating material. It consists of a closed vessel having a vapour inlet and outlet and a number of conduits extending through the vessel with walls pervious to the vapours and impervious to the solid treating material. Each conduit has a vapour-tight hopper for supplying active treating material and receiving spent material. The treating material used depends on the treatment to which it is desired to subject the vapour. Fuller's earth, bauxite, bentonite or silica gel can be used to polymerise unstable, unsaturated hydrocarbons in cracked distillates or metallic oxides such as copper oxide to remove contaminating sulphur compounds. W. S. E. C.

1414. Purification of Liquids. C. J. Rodman and R. P. Dumise. Assrs. to Buckeye Twist Drill Co. U.S.P. 1,824,498, 22.9.31. Appl., 25.6.29.

A process for removing high vapour pressure fluids from a relatively low vapour pressure fluid is described. The liquid mixture is sprayed under super atmospheric pressure into a vacuum of 28-30 in. Hg., against baffles to release the high vapour pressure fluids from the low vapour pressure fluids. The baffles are arranged in staggered relation so as to arrest the oil and cause the moisture and volatiles to be separated and the oil can then be collected. W. D. S.

1415. Separating Alcohols from Oil. T. A. Werkenthin and Solar Refining Co. U.S.P. 1,824,521, 22.9.31. Appl., 24.11.25.

Small amounts of alcohol are removed from petroleum still residues without detrimental results to the petroleum. The liquid is heated to 220-250° F. at normal pressures and the alcohol withdrawn. The heated mixture is then finely divided and maintained at 220-250° F. under reduced pressure thereby withdrawing the last traces of alcohol. W. D. S.

1416. Sulphonating Mineral Oil. L. D. Myers and Twitchell Process Co. U.S.P. 1,824,615. 22.9.31. Appl., 2.3.29.

A process is described whereby medicinal white oil, light coloured technical oils and mineral oil sulphonates are produced. The latter may be mahogany sulphonates for use as saponifiers in the soap, stearic acid and glycerine

industries, or sludge layer sulphonates for decomposing or resolving the petroleum and water emulsions encountered in oil fields. The sulphonation is carried out with a fuming H_2SO_4 containing 25 to 35% free SO_3, at a temperature of 50° to 60° C. W. D. S.

1417. Fractionation of Certain Hydrocarbons. H. S. Davies and W. J. Murray, Assrs. to Petroleum Chemical Corpn. U.S.P. 1,824,947, 29.9.31. Appl., 5.8.26.

The fractionation equipment described consists of tar separators, rectifying columns, and fractional condensers, and is divided into two halves, the first dealing with the liquid products and the second with the gases obtained by stripping the absorption oil from the absorber installed between the two halves. It is claimed that close cut products are obtained comprising: (1) gaseous, containing ethylene, propylene, methane and hydrogen; (2) liquid (under slight pressure) containing butylene, amylenes and hexylenes; (3) crude naphtha suitable for motor fuel; (4) cycle stock; (5) fuel oil. H. C. R.

1418. Treating Hydrocarbons. V. N. Jenkins and C. P. Wilson, Junr., Assrs. to Texas Co. U.S.P. 1,825,270, 29.9.31. Appl., 14.6.24.

In the treatment of heavy hydrocarbon oils with aluminium chloride for the purpose of producing lower boiling hydrocarbons, the volatilisation of the halide with consequent choking of vapour lines may be avoided by first treating the hydrocarbon and aluminium chloride below the conversion temperature with hydrogen chloride to form a liquid treating agent and then, after heating to the conversion temperature, carrying out the reaction in the presence of hydrogen chloride gas. In this way volatilisation of the aluminium chloride is prevented while the treating agent is maintained in a reactive condition. Furthermore the operation of the still is more even and a higher yield of low boiling products is obtained. The use of dry hydrogen chloride is unnecessary as any water present reacts with the aluminium chloride to produce more hydrogen chloride. Suitable plant is described for carrying out the process. S. F. B.

1419. Treating Hydrocarbons. E. R. Wolcott, Assr. to Texas Co. U.S.P. 1,825,294, 29.9.31. Appl., 14.6.24.

A suitable form of apparatus for carrying out the treatment of heavy hydrocarbons with aluminium chloride in the presence of hydrogen chloride (see preceding abstract) is described. The apparatus consists of a still with inlets at the top for oil and aluminium chloride (or the liquid treating reagent produced from the oil, aluminium chloride and hydrogen chloride) with a tube for hydrogen passing to the bottom of the still suitably perforated to allow even distribution of the gas. A run-off tube is provided while a vapour line at the top passes through a dephlegmator to a condenser. Stirring, scraping or agitation devices can be incorporated if desired. S. F. B.

1420. Decolourising Oils by Filtration through Paper. W. S. Baylis and Filtrol Co. U.S.P. 1,825,300, 29.9.31. Appl., 24.4.28.

Petroleum distillate is purified and decolourised by agitating with 1% by weight of strong H_2SO_4 (added slowly) and then filtering the mixture through filtering paper containing acid activated clay having a free sulphuric acid content. The clay is incorporated in the paper during manufacture to the extent of 10–25% of its finished weight. A special clay ("Filtrol") which has 0·15–0·50% of free H_2SO_4 uniformly distributed, is used. W. D. S.

Cracking.

1421. Oil Treatment. Petroleum Conversion Corpn. E.P. 357,265, 24.9.31. Appl., 24.6.30.

In the process of cracking or converting hydrocarbon oils in the vapour phase by the use of a gaseous heat carrier, corrosion can be substantially reduced by keeping the sulphur content of the mixed gases and vapours below a predetermined value. This value is to some extent determined by the cracking stock employed, but is usually in the neighbourhood of 1%. Care must be taken that the carrier gas used contains no oxidising agents capable of converting any sulphur present into sulphur dioxide or trioxide, as these gases are highly corrosive. Reference is made to E.P. 336,380.

S. F. B.

1422. Converting Hydrocarbons. C. P. Dubbs. U.S.P. 1,820,057, 25.8.31. Appl., 30.6.18. Divided, 18.6.31.

The object of the invention is to provide a process in which the oil is passed in small streams through a cracking zone into a large chamber located outside the fire zone; thus the use of relatively small pipes in the fire zone is possible, and in which deposition of carbon is substantially eliminated. The precipitated carbon is deposited in that part of the apparatus outside the fire zone.

R. P.

1423. Apparatus for Treating Oils. C. P. Dubbs. U.S.P. 1,820,125, 25.8.31. Appl., 27.6.21. Renewed, 17.1.29.

The invention relates to improvements in processes and apparatus for cracking oils, especially to a process in which the pressure in the separate units may be controlled accurately by means of a single pressure relief valve in a common receiver.

The raw oil is pumped into a main line placed above the dephlegmators, any number of units may be connected up to form a battery of stills. The cool oil is introduced into the top of the dephlegmators and serves as a refluxing medium for the vapours. The mixture of raw oil and distillate is drawn off from the bottom of the dephlegmators and charged to the heating tubes, where, after being heated to a cracking temperature, it is passed into vapour chambers situated above the furnace. The vapours rise through vapour lines and are refluxed in the dephlegmating columns.

The unvaporised oil from the vapour chambers is drawn off and directed to a residuum distillation chamber where it is subjected to reduced pressure, and the distillate obtained may be returned as feed oil through the dephlegmators. The difference in pressure between the two stages can be accurately controlled by a valve situated in the draw off lines. Instead of passing the residuum to the secondary stage it may be directed into the main residuum line, whence it flows to storage.

R. P.

1424. Apparatus for Treating Hydrocarbon Vapours. W. F. Sims and V. U. Cloer. U.S.P. 1,821,116, 1.8.31. Appl., 7.5.29.

By cracking, fractionating, and cooling under a vacuum it is claimed that the yield of products containing unsaturated and aromatic compounds can be increased. An apparatus is claimed, comprising a preheater with means for heating and for maintaining pressure, a flashpot, a cracking still, a plurality of fractionating towers, a cooler and separator, a suction pump to produce suction through the system and a subatmospheric pressure on cracking still and flashpot; an oil pump, with a feed pipe connected to the

output of the last fractionating tower; and a pressure pipe through the several fractionating towers to the flash pot whereby suction is exerted forward through the system and pressure backward through the feed until released into the catchpot. R. P.

1425. Apparatus for Heating and Mingling Fluids. C. P. Tolman. U.S.P. 1,821,333, 1.9.31. Appl., 3.1.27.

The invention relates to the heating of fluids by mingling other heated fluids in order to provide a method wherein the fluid to be heated may receive the desired heat content within a minimum temperature gradient and within a minimum time.

In the cracking of petroleum it is of great importance that the heat transfer takes place within a narrow temperature range. By the operation of the invention the hydrocarbon vapour to be cracked will be so intimately mixed with the hot fluid that the cracking temperature will be attained in less than the reaction time, so that intermediate products will not be formed.

To carry out the process a carrier gas is heated in a blast stove and injected into chambers into which hydrocarbon vapours are introduced. R. P.

1426. Cracking Oil. G. Egloff, Assr. to Universal Oil Products Co. U.S.P. 1,821,746–50, 1.9.31. Appl., 6.12.20; 9.12.30; 21.12.25; 7.12.25.

These relate to a cracking process wherein charging stock, residuum and recycle oil are separately heated to their correct temperatures in superimposed combustion chambers. They are then delivered to a common expansion chamber maintained under reduced pressure, and from thence to a dephlegmator, condenser and receiving apparatus. Heat exchange between bottoms and raw material is provided for. Diagrams and operation details are appended. H. C. R.

1427. Treatment of Hydrocarbons. J. C. Clancy. U.S.P. 1,822,127, 8.8.31. Appl., 29.4.30.

The present invention is based on the discovery that an alkaline substance such as sodium carbonate in a fused mixture with alkaline metal cyanide has the effect of preventing the evolution of hydrocyanic gas when used as a catalyst in cracking.

A process is claimed whereby oil is vaporised and cracked in presence of a cyanide salt, in a molten mixture with another salt which is capable of preventing the decomposition products liberating hydrocyanic acid.

By cracking in the presence of the cyanide catalyst a higher aromatic content in the cracked gasoline is claimed. Desulphurisation also is claimed. R. P.

1428. Converting Hydrocarbons. C. W. Watson. U.S.P. 1,823,922, 22.9.31. Appl., 26.6.28.

The invention relates to cracking processes wherein reflux condensate is returned to the cracking zone. A process is claimed wherein a high boiling oil is heated to conversion temperature and vapours produced are passed to a fractionating column. The condensate is returned to the cracking coil and the vapours to a second column to eliminate further condensate from the system. Partially heated oil is withdrawn from the heating coil in heat exchange with the condensate in the bottom of the dephlegmating towers to reboil the condensates.

The oil thus heated is returned for further cracking and the vapours from the second fractionating tower are condensed to form the desired final distillate. R. P.

1429. Treating Hydrocarbons. L. Kirschbraun. U.S.P. 1,824,215, 22.9.31. Appl., 1.11.22. Renewed, 25.11.29.

A continuous process is claimed wherein the oil is raised to cracking temperature in a coil, passed to a reaction chamber of special design, through which the oil is caused to flow downwardly and undergo substantial vaporisation.

The vapours released rise in an annular stream through the reaction zone out of contact with the downflowing oil, a part is condensed and returned to feed, whilst the uncondensed vapours are collected as final product. R. P.

1430. Conversion of Petroleum Oils. R. Cross. U.S.P. 1,825,337, 29.9.31. Appl., 22.7.22.

Oil is preheated and circulated through a heating zone where it is raised to cracking temperature, thence directed into a large chamber in which a body of oil is allowed to collect and there undergo molecular change while maintained under pressure. In this reaction chamber digestion takes place and low boiling oils are formed. The synthetic crude while in the heated condition and under pressure is passed through a coil in a container through which the incoming oil is circulated and preheated. R. P.

1431. Converting Hydrocarbons. W. M. Cross, Assr. to Gasoline Products Co. U.S.P. 1,825,338. 29.9.31. Appl., 1.2.26.

This invention relates to a cracking process in which a forced draught of air through a heat exchanger situate in the dephlegmator is preheated and used for the main furnace air duct. Details of the construction of the heat exchanger are given and consists essentially of a plurality of sets of corrugated sheets. H. C. R.

1432. Valve. R. E. Wilson, Assr. to Standard Oil Co. (Ind.). U.S.P. 1,825,378, 29.9.31. Appl., 27.4.26.

This invention relates to a needle-type valve suitable for controlling the flow of partially cracked petroleum products without the disadvantage of carbon depositing in or beyond the valve. H. C. R.

Hydrogenation.

1433. Progress and Prospects of Hydrogenation. E. F. Armstrong. *Gas World, Coking Section*, 1931 (2466), 22.

The conversion of coal into oil carries with it the problem of the conversion of the primary tar oil into a product suitable for use as a motor spirit, which might be solved by cracking or catalytic hydrogenation. The tendency in Germany recently has been to obtain the oil from the coal by a low temperature carbonisation process, and then to hydrogenate this by a modified Bergius process.

G. T. Morgan, working with low temperature tar, topped at 200° C. with molybdic acid-sulphur catalysts and pressures of 120–200 atm., has obtained promising results. As catalysts for coal hydrogenation the patent literature shows that oxides of iron, cobalt and nickel, containing 0·4 to 0·6% of alkali, have been claimed, in addition to catalysts containing copper, zinc, cadmium, boron, aluminium, silicon, vanadium or their compounds, for use at elevated temperatures and pressures. The main functions of the catalyst are to

present a large surface per unit mass, a strong specific absorption for the reactants, and the ability to form loose transient addition compounds with the interactants.

The methods of hydrogen production on a large scale are discussed, and it is suggested that the main power stations in the grid scheme could produce hydrogen electrolytically as an off-peak load, thereby making cheap hydrogen and reducing costs for power production at the same time. Coke oven gas as a source of hydrogen is discussed with reference to the Bronn-Linde and Claude systems, which involve compression to 12 atm., the removal of the carbon dioxide by water and alkali washing under pressure, followed by fractional cooling to remove unsaturateds, methane and finally, carbon monoxide, the final traces of which can be eliminated by means of irrigation with liquid nitrogen at a low temperature. The hydrogen so obtained is of good purity and of approximately the same cost as that made by the direct purification of water gas.

The author urges more co-operation between the collieries, the gas companies, the coke oven companies, and the tar distillers, and suggests that the collieries represent the obvious location for hydrogenation plant.

W. E. J. B.

1434. Low-boiling Hydrocarbons from Tars, Oils and the Like. E. G. Brown, H. W. Strong and the I. C. I., Ltd. E.P. 357,589, 25.9.31. Appl., 25.6.30.

Tars, phenols and hydrocarbon oils may be hydrogenated to produce low-boiling hydrocarbon fuels in the vapour phase, using magnetic iron oxide, or the product resulting when this oxide is reduced in an atmosphere of hydrogen, as catalyst. Small amounts of tin or metals of Groups V and VI of the Periodic Table may be added to the catalyst with advantage. The reaction is effected at temperatures between 400–600° C., and under a total pressure of 200 atm. or higher. S. F. B.

1435. Hydrogenating Heavy Organic Compounds to Produce Light Hydrocarbons. J. M. F. D. Florentin and A. J. Kling. E.P. 357,822, 1.10.31. Appl., 27.10.30.

The method consists in compressing organic materials to high pressure, and heating to a temperature higher than the lowest point in the range at which decomposition commences. The material is then admitted into the reaction chamber in presence of hydrogen and catalyst under a pressure of from 200 to 300 atm. at a temperature lying between 380° and 500° C.

Thus if hydrogenation is carried out at 250 atm. and 450° C., it will be convenient to raise the liquid to be hydrogenated to a temperature of 450–480° C. and to a pressure of 500 atm. R. P.

Chemistry of Petroleum.

1436. Oxidation of Methane. II. The Homogeneous Oxidation of Methane under High Pressures. K. Yoshikawa. *Bull. Inst. Phys. Chem. Res.*, Japan, 1931, **10**, 305–15.

Little work has been reported on the oxidation of methane under high pressures except the experiments on explosion. The present experiments were carried out by a flow method at 12–100 atm. with mixtures of methane and air in the ratio of 1 : 1, 2 : 1 and 3 : 1. Within this range of pressures, the reaction occurred rapidly at 300–400°, CO, CO_2, H_2O, CH_3OH and HCHO

being found chiefly as reaction products; an increase in the pressure favoured the formation of CH_3OH. By enlarging the surface of the reaction vessel the reaction is choked, an indication that the main reaction is a homogeneous chain-type reaction; when the reaction is choked in this way, the concentration of CH_3OH and HCHO in the condensation product is increased. The effects of pro- and anti-knock materials upon the rate of reaction were studied by adding them to the gas mixture of methane and air (3 : 1), which reacted under 25 atm. at 300°, 350° and 400°. A little nitric acid vapour added to the gas mixture greatly promotes the reaction at 350°, while the presence of lead tetraethyl vapour in a small quantity retards it. Little change in the composition of the condensation product is observed on the addition of pro- or anti-knock material. W. S. E. C.

1437. Isolation and Determination of the Cyclohexane in a Mid-Continent Petroleum. J. H. Bruun and M. M. Hicks-Bruun. *Bur. Stand. J. Res.*, 1931, **7**, 607–615.

The Mid-Continent Crude was fractionated by distillation into 1° C. cuts and the fraction 74° C.–85° C. nitrated to remove benzene.

Further distillation of this fraction was then carried out when most of the material concentrated in a cut 80° C.–80·5° C., and the cyclohexane was separated from this by systematic fractional melting, yielding a very pure product, 99·96 mol. %.

The boiling point, freezing point, specific gravity, refractive index, molecular weight and critical solution temperature with aniline were determined on the isolated hydrocarbon, and were found to give good agreement with similar data obtained on synthetic cyclohexane.

Photographs of the visible, ultra violet and infra-red absorption spectrum were also taken.

The petroleum fraction 80° C.–80·5° C. was found to contain 94 mol. % of cyclohexane, giving a calculated content of 0·33% of the hydrocarbon on the crude petroleum. D. L. S.

1438. New Derivatives of 2-methylbutane. W. R. Forsman. *Finska. Kem. Med.*, 1931, **40**, 47–8.

Isoprene alcohol was formed by treating isoprene hydrobromide with silver hydroxide. The alcohol gives isoprene at 335° on thorium oxide. The acetate and stearate were prepared from the dichloride and the dibromide of the alcohol. Treatment with alkali gave epichloro- and epibromohydrin. Isoprene alcohol with hydrogen bromide gave a monobromohydrin, and with hydrochloric acid a chlorobromohydrin. From dimethylglycerol the diacetate, diproprionate, dibutyrate and dibenzoate were prepared by treatment with pyridine and the acid chloride or anhydride. By metathesis of isoprene alcohol dibromide with silver acetate the diacetate was also prepared. Isoprene hydrobromide and silver thiocyanate gave a diallyl mustard oil, which reacted with bromine and reduced to isoprenеamine by treatment with hydrochloric acid. W. S. E. C.

1439. Thermal Behaviour of Sulphur Compounds in Hydrocarbon Solvents: A. Aliphatic Mercaptans. W. M. Malisoff and E. M. Marks. *Ind. Eng. Chem.*, 1931, **23**, 1114–1120.

The authors have examined the behaviour of seven mercaptans at 300° C., 425° C. and 475° C. in hydrocarbon solution at several dilutions. The solvents chosen, namely benzene, *n*-heptane, 2·2·4-trimethylpentane and straight-run naphthas, were shown to be sufficiently stable for the purpose, no appreciable reaction taking place either with the mercaptans or with their decomposition

products at temperatures below 475° C. In analysing the products a modification of the method for estimating mercaptans described by Borgstrom and Reid (*Ind. Eng. Chem.*, Anal. Ed., 1929, **1**, 186) was used.

The decomposition of the aliphatic mercaptans was found to be homogeneous in the gas phase up to 475° C. apparently taking place with the formation of hydrogen sulphide and an olefine. The decomposition of amyl mercaptan, while being to some extent depressed by the presence of carbon dioxide or hydrogen, is promoted by benzene as compared with its behaviour in naphtha. It is, of course, possible that the naphtha itself may aid in promoting the reaction. For purely aromatic types of mercaptans the contrary appears to be true. Benzyl mercaptan is less stable than the lower members of the aliphatic series; although the branched chain isomers are less markedly stable than the straight chain compounds at 475° C., the higher members are probably less stable than the lower although this is by no means certain.

It is not possible to say from the order of accuracy of the data available whether the reaction is of the first or second order. The decomposition points to a reaction of the first order, and this is confirmed by the approximate independence of the rate of decomposition and of the concentration; furthermore, tests for a bimolecular reaction give less satisfactory agreement.

A rough estimate of the temperature co-efficient for a 50° C. range leads to a value of 5·0 for *n*-amyl mercaptan, assuming unimolecular constants. This corresponds to a temperature co-efficient for 10° C. intervals of 1·38 and to a heat of activation of 33,500 cals. The value for $\frac{E}{RT}$ for a velocity constant of 0·00158 sec.$^{-1}$ is estimated at 24·7. S. F. B.

1440. Exothermic Reactions at High Temperatures. D. Binnie and T. S. Wheeler. *J.S.C.I.*, 1931, **50**, 418T.

This paper describes the removal of oxygen from air by means of hydrogen, the production of hydrogen chloride, and the interaction of hydrogen and chlorine in the presence of methane.

This latter process utilises the exothermic nature of the hydrogen-chlorine reaction to energise the transformation of methane into acetylene, about 28% of the CH_4 supplied being converted into acetylene under optimum conditions.

The application of the above principle offers possibilities in industrial practice. W. E. J. B.

1441. Preparation and Physical Properties of Ethyl and Methyl Acetylene. F. R. Morehouse and O. Maass. *Can. J. Research*, 1931, **5**, 306–312.

The hydrocarbons were prepared by the action of the corresponding halides upon sodium acetylide dissolved in ammonia, the general method of procedure being modified in certain details. The removal of ammonia from the hydrocarbons was conveniently carried out by passing them through water maintained slightly acid by the addition of sulphuric acid. This procedure was found to give better yields than when water alone was used for the purpose without in any way affecting the hydrocarbons. It was also found that the gases could be dried over phosphorus pentoxide, which does not appear to exert any polymerising action. Physical properties including the boiling and melting points are given, while the vapour pressures, densities and surface tensions are reported for a considerable temperature range. These properties, together with constants calculated from the data, are compared with similar data for olefines and paraffins. S. F. B.

1442. Decomposition of Sodium Hypochlorite: An Ion Reaction. W. F. Underwood and E. Mack, Jr. *J. Physical Chem.*, 1931, **35**, 2650–7.

The rate of decomposition of aqueous sodium hypochlorite solutions practically free of traces of heavy metals has been studied at 45° in the presence of various salts. On the assumption that the chemical reaction is $2\ ClO^- \rightarrow 2\ Cl^- + O_2$, a positive salt effect was observed, and the slope of the plot of log k_0 against $\sqrt{\mu}$, where k is the velocity constant and μ the ionic strength of the solution, is + 1, as predicted from Brönsted's theory.

The velocity data show that the reaction is kinetically bimolecular in strong salt solutions. W. D. S.

1443. Reaction Velocity of Sulphuric Acid with Unsaturated Hydrocarbons. M. B. Marcovitch and B. G. Moore. *Nat. Petr. News.*, **23** (41), 33–38; (42), 27–32.

The reaction velocity of H_2SO_4 with unsaturated hydrocarbons of the type occurring in cracking still gases is studied. In particular propylene, the butylenes and butadiene are examined. The reaction velocity is determined by following the change of volume per unit of time during non-turbulent contact with sulphuric acid in an apparatus of the Orsatt type. Absorption is conducted at constant volume and temperature of 18·5° C. — 22°C. with acid containing 63%, 68%, 75%, 77% and 83% H_2SO_4. The gases are diluted with air to a concentration of about 60% gas before examination, while the results are calculated to indicate the average velocity of absorption at different concentrations of gas.

Experimental work designed to test the reliability of the data for analytical purposes is described and a method for the partial corrections of results given. The application of the method appears to be limited. H. G.

1444. Acetyl Value of Oxidised Mineral Oils. T. Yamada. *J.S.C.I.* (Japan), 1931, **34**, 188–9.

Samples of technical white oil (paraffin base), transformer oil (naphthene base), cracked oil and a less refined switch oil were oxidised in the open air at 130°. The results, which are given in tables and graphs, show that, when oxidised, refined oil shows acid and saponification values and saponification of the acetylated product increasing at a nearly definite ratio. The unsaponifiable portions have a distinct apex on the curve of their acetyl value. Alcohol and acid formation is rather small in the case of the less refined oil.

W. S. E. C.

Analysis and Testing.

1445. Adhesion of Lubricating Oil Films to Metallic Surfaces. A. Boutaric and R. Amiot. *Ann. des Comb. Liquides*, 1931, **6**, 443–458.

A heart-shaped plate was immersed in oil, withdrawn and allowed to drain (point downwards) at constant temperature while being suspended from a spring balance. The variation weight of adhering oil diminished with time, and eventually a steady value (π) was reached. The value of (π) was a function of the viscosity of the oil, and was slightly greater for vegetable oils as against mineral oils of equal viscosity.

Similar conclusions were drawn from other experiments in which a U-shaped plate was centrifuged after immersion in the oil and weighed at fixed intervals. This quantity π varies according to the nature of the metal, and also increases with the acidity (or age) of vegetable oils when the metal concerned is iron or copper. Aluminium and stainless steel did not differentiate between vegetable oils of varying acidities. Hence the different behaviours of metals is attributed to chemical attack of iron and copper by the acid content of the oil. C. I. K.

1446. Still Head for Laboratory Columns. E. B. Kester and R. Andrews. *Ind. Eng. Chem. Anal. Edn.*, 1931, **3**, 373.

The still head is constructed of pyrex glass and is attached to the column by a ground glass joint.

Vapours pass up the column and enter a reflux condenser to be totally condensed. The condensate returns down the condenser, where any desired portion may be returned to the column as reflux by adjusting the volume to be withdrawn from the system. A. R. S.

1447. Analysis of Gaseous Hydrocarbons. R. Rosen and A. S. Robertson. *Ind. Eng. Chem., Anal. Edn.*, 1931, **3**, 284.

As a result of a study of a number of synthetic mixtures of gaseous hydrocarbons a master graph is presented whereby the analysis of certain gaseous hydrocarbon mixtures, *i.e.*, refinery gases may be completed in 60 to 75 mins., using only 200 to 250 c.c. of liquid nitrogen.

The apparatus consists essentially of two calibrated bulbs, between which are two manometers, one open to the atmosphere, the other, of the closed limb type, open to the second of the calibrated bulbs, which acts as a distillate receiver. Between the first bulb or sample holder and the manometers is a glass bottle or still with a thermocouple inserted.

Fixed gases are removed while the sample is held at the temperature of boiling nitrogen by means of a vacuum pump. The receiver is then allowed to warm up at controlled rate and the temperature percentage distillate curve got by simultaneous readings of pressure on closed manometer and millivoltmeter. During the operation the apparatus is held at 760 m.m. pressure for convenience of correlation of results.

Results of analyses using this method against the Podbeilniak apparatus are given, the agreement being close. A. R. S.

1448. Determination of Olefine, Aromatic and Paraffin Hydrocarbons in Neutral Oil from Coal Tar. E. B. Kester and W. D. Pohle. *Ind. Eng. Chem., Anal. Ed.*, 1931, **3**, 295.

The oil sample is shaken with three volumes of 80% sulphuric acid and reduction in volume R_1 is recorded. The oil is then distilled to an end point 5° higher than its original end point and the reduction in volume from distillation R_2 is recorded after a small correction, which may be considered a constant over the same boiling ranges. From the sum of $R_2 + R_1$ the olefine percentage is calculated.

The distillate or an aliquot part of it is then shaken with 3 volumes of 98% sulphuric acid, and the contraction noted after deduction of a factor for the solubility of paraffins in 98% acid. The corrected value r_3 is then computed to the whole oil basis by equation

$$R_3 = \frac{r_3 \times (\text{orig. vol.} - R_1 - R_2)}{V_3}$$

where V_3 is volume of oil used for aromatic determination the original volume being 100 and R_3 the volume of aromatics in the original sample. A. R. S.

1449. Development of Gasoline Specifications. C. K. Francis. *Oil and Gas J.*, 1931, **30**, 23, 30.

A short résumé is given of the history of gasoline production and of the influence which automobile development has had on the production in regard to both quality and quantity. This is followed by a history of the methods which have been employed in testing the quality of gasoline, the endeavours made to relate the results of these tests with engine performance, and the growth and development of specifications designed to differentiate gasolines on a quality basis. The old designations based on gravity limits are given in detail and the gradual change to the use of distillation methods as a measure of volatility, etc., outlined. The evolution of the distillation test method is described in detail and information given on the influence of the position of the thermometer in the distillation flask on the results obtained. Past and present U.S. Bureau of Mines Specifications are quoted and the gradual elimination of relatively unimportant tests, such as gravity, colour, I.B.P. and the inclusion of more informative tests demonstrated. Reference is made to the improvement effected in antiknock value of gasoline by means of crude selection and improved refinery methods. The omission of reference to this property in standard specifications is pointed out. R. A. E.

1450. Exhaust Gas Analysis. C. B. Dicksee. *Auto. Engr.*, 1931, 285, 401-406.

The author deals with the use of the Orsat flue gas analysis apparatus for the analysis of the exhaust gases of I.C. engines. The method of using the apparatus, the necessary combustion equations and the method of applying them are described.

From gas analysis of petrol engine exhausts the mixture strength and volumetric efficiency can be determined, and the distribution of the charge in multi-cylinder engines, and the heat balance, can be investigated. The loss of charge in 2-str. petrol engines can also be determined. The actual amount of air used in compression-ignition engines can also be determined by exhaust gas analysis and not by any other method.

Tables and curves compiled from results of actual analyses of exhaust gases of various types of I.C. engines are given to illustrate these points. J. M. F.

See also Abstract No. 1453.

Anti-Detonation.

1451. Photographic Method of Determining the Resistance of Gasoline to Detonation. M. Aubert and R. Duchêne. *Compt. Rend.*, 1931, **192**, 1633-5.

A series of photographs is taken that show the flame propagation through a fuel-air mixture under a 7 : 1 compression. The temperature is raised slowly until the flame propagation becomes instantaneous. This temperature is considered as the detonation index of the fuel. The data corroborate actual engine tests and also the known antidetonating properties of the lead tetraethyl. W. S. E. C.

1452. Rationale of the Movement of Flame through Explosive Mixtures of Gases: I. Explosions in Closed Containers. O. C. de C. Ellis. *J.S.C.I.*, 1931, **50**, 403T-409T.

Reference is made to the apparatus used for a photographic study of flames—described in Safety in Mines Research Board Paper No. 32.

Considering the gas mixture as a number of thin spherical layers concentric about the point of ignition, contained in a closed sphere concentric with the layers, the pressure is highest at the flame surface and grades smoothly inwards without discontinuity. Outwards, the pressure also grades smoothly, but is discontinuous since the outer surface of the luminous layer is a surface of discontinuity. In some mixtures, *e.g.*, those containing a low proportion of inert gas, the pressure at the source reaches a critical value when the rate at which energy is liberated enables the flame to propagate itself as a shock wave—giving rise to detonation.

Since the gas outside the flame surface is moving inwards, two winds are set up and the division of force emitted by the expanding layer between the inner and outer gases is approximately as their volumes. A critical point is reached when the two volumes are equal and at this point the rate of change of force is a maximum and the expansion of the flame appears to suffer an arrest. When the layer next the wall of the vessel is luminous, the pressure in the vessel is at a maximum and there is only one wind—an inward surge. The latter has continuously and increasingly induced chemical action in the gases undergoing recompression—thereby increasing their temperatures and producing luminance known as "after-burning."

When the vessel is not a sphere or ignition is not central, some parts of the flame reach the walls before others, and the proportion of energy supplied to the inner gases from the expanding layer is increased at the expense of the outer gases—the flame surface appearing to trip or halt or retreat. These points are illustrated by photographs of mixtures of CO and O_2, taken after certain intervals of time after ignition. The outward surge is the agent by which the flame is shaped, and when ignition is not central the surge is not the same in all directions and the flame adapts itself to the shape of the container. This again is illustrated by photographs.

When two or more flames are started simultaneously and symmetrically in a closed tube—nodal planes are set up at the points of meeting of the outward surges. When synchronous ignitions are asymmetric, however, each flame continually centres itself and the boundaries of the fields continually shift towards equality. Further photographs illustrate this. The latter show that the flame surface has taken the form of a vortex.

If the explosive mixture is in more or less violent motion when ignition occurs the flame tends to become formless and atomised, the area of the flame surface being increased by the motion of the gas. Hence turbulence tends to shorten the flame period. C. C.

1453. Oxidation and Ignition of Gaseous Mixtures. M. Prettre. *Ann. des Comb. Liquides*, 1931, **6**, 7-42, 269-309, 533-558.

An apparatus is described, based on the "pyrometer" method previously used by Mallard and Le Chatelier, Taffanel and Le Floch, and Mason and Wheeler, for the accurate determination of the ignition temperatures of inflammable gaseous mixtures. In this apparatus, mixtures of hydrogen and air, hydrogen and oxygen with varying proportions of nitrogen, argon and carbon dioxide, carbon monoxide and air and carbon disulphide and air have been studied. In all these cases the ignition temperature increases rapidly when the concentration of the combustible gas exceeds the upper limit of inflammability at ordinary temperatures. Below this limit the

variation in ignition temperature is much less, being practically constant in the cases of mixtures of hydrogen, oxygen and carbon dioxide, and of carbon monoxide and air.

A small amount of burnt gases remaining in the combustion vessel markedly raises the ignition temperatures of mixtures containing hydrogen, lowers that of mixtures of carbon monoxide and does not effect carbon disulphide-air mixtures. In the cases of mixtures, except those of hydrogen, when the combustion vessel is only slightly above the ignition temperature there is a lag of several seconds before ignition. During this period slow oxidation occurs and the gases become luminous, the luminosity increasing until the flame appears. If the temperature of the combustion-vessel is slightly below the ignition temperature the mixture also becomes luminous and remains so for several minutes. Further experiments on this phenomenon of luminosity showed that it was most intense when the mixture contained insufficient air for complete combustion. With carbon disulphide-air mixtures at temperatures a little below that at which luminosity appeared, a brown deposit was obtained which was shown to be carbon monosulphide. With mixtures of paraffin-hydrocarbons and air, the luminosity was observed at 220–250°, the ignition occurring much later—about 500° C. or higher. The behaviour is dependent upon the concentration. With excess of air no ignition occurs until above 600° C. When insufficient air is present for complete combustion, a much more intense luminosity occurs. About 30° C. or 40° C. above the temperature of luminosity, a flame occurs (first ignition). If the temperature is about 20° C. higher, inflammation does not occur, but simply slow, luminous combustion. Above 650° C. ignition again occurs (second ignition). The first ignition only occurs if the gas has been some time in the combustion vessel and is probably caused by an intermediate product of combustion. These results explain the low ignition-temperatures obtained by methods involving progressive heating of the mixture. The first type of ignition is completely suppressed by appropriate quantities of anti-detonants such as benzene or lead-tetraethyl. It is this first ignition which gives rise to the phenomenon of knocking in engines. Anti-detonants, by suppressing this, enable combustion to occur in the normal way and at the normal temperatures. A historical review and extensive bibliography are also given. E. B. E.

Engines.

1454. Design of Injection Nozzles. Summary of five N.A.C.A. reports. *Aircraft Eng.*, 1931, **3**, 281–284.

No. 382.—*Basic requirements of Fuel Injection Nozzles for quiescent combustion chambers.*—A report dealing with experiments carried out on a single-cylinder engine to determine the best design of multi-hole nozzles for this type of engine. The findings are not conclusive, but certain well defined basic requirements become apparent, and are fully described.

No. 369.—*Effect of Orifice length/diam. ratio on the co-efficient of discharge of Fuel Injection Nozzles.*—The report describes experiments with three nozzles, viz., one of constant diameter, another with a plain stem, and a third with a helically-grooved stem. A wide range of injection pressures and length/diam. ratios are investigated with fairly conclusive results.

No. 372.—*Development of an Impinging Jet Fuel Injection Valve Nozzle.*—An investigation of the possibilities and limitations of the two-cycle high speed fuel injection engine as a means for increasing the power per unit of displacement in an engine for aircraft use.

The main difficulty encountered was the design of a suitable nozzle, and the nozzle adopted is fully described and spray photographs are given showing its performance.

No. 373.—*Investigation of the discharge rate of the Fuel Injection system.*—A detailed examination of the fuel flow per degree of crank angle from a multi-hole nozzle. Interesting results were obtained showing that the useful injection takes place over a third only of the total injection period under the conditions described.

No. 376.—*Effect of high air velocities on the distribution and penetration of a Fuel Spray.*—An investigation showed that air velocities of 400 ft./sec. were necessary to break up the central bore of a fuel spray, and that air directed counter to the spray was much more effective in this respect. C. H. S.

1455. Co-ordination of Engine and Carburettor. O. H. Ensign. *J.S.A.E.*, **29**, 192–197.

The author gives a résumé of the earlier types of carburettor, and the reasons for their modification to the present general type, the chief reason being the alteration in the volatility of fuels universally obtainable to-day.

The need for extra "accelerating" fuel at present is explained by the fact that gasolines are now relatively very much heavier, and consequently the difficulty in getting sufficient fuel into the air stream when accelerating is very much greater. It is pointed out that manifolds cannot be kept hot enough to vapourise present fuels in sufficient quantity to meet all conditions without sacrificing volumetric efficiency and consequently power output.

The forms of present so-called economisers are dealt with, which tend to correct the tendency of carburettors to give over-rich mixture at the higher velocities. Curves showing power output on various engines and with varying fuel consumptions are given, showing the advantages of the economiser. From these curves it appears evident that it is much more economical to obtain a given horse-power on full throttle at low speed, than to develop the same power at a higher speed on part throttle. Stress is laid on the effect of gear ratio on economy, and the author is of opinion that the ideal to be striven for is a transmission system that will cause the engine speed to fall as power requirements fall, thus ensuring the engine being run at high torque at all loads with resulting economy as previously explained. C. H. S.

1456. Air-Cooled Cylinder-Head Design. R. Chilton. *J.S.A.E.*, **29**, 185–191.

Efficient air-cooling depends upon adequate conductivity from the regions of greatest heat, and the degree of cooling of the conductors (fins) by the air flow.

The tendency of a fuel to detonate limits the possible output of an engine, and the importance of the "Octane Number" of fuels is stressed. The author states that if fuels of 80 Octane No. were universally obtainable, the commercial rating of an engine could be raised by 30%, where the present rating has to be based on the worst current fuels of approximately 53 Octane No.

The author is in favour of fitting thermocouples to cylinder-heads, and of training pilots to use same as an indicator of permissible throttle opening, which would make sure that the engine was being run below the temperature at which dangerous conditions develop. C. H. S.

1457. FACTORS CONTROLLING ENGINE-CARBON FORMATION. W. H. Bahlke, D. P. Barnard, J. O. Eisinger, and O. Fitz Simons. *J.S.A.E.*, 1931, **29**, 215-222.

Improved temperature regulation, piston ring design, etc., have minimised carbon formation in recent years, but combustion chamber carbon appears to offer an obstacle to the use of appreciably high compression ratios.

The Conradson carbon residue of an oil has been assumed to measure carbonizing tendency, but recent work has emphasised the probable dependence of carbon formation on the volatility of the oil.

Preliminary work on a 2$\frac{1}{16}$ in. by 4$\frac{1}{2}$ in. six-cylinder sleeve-valve engine (34 b.h.p. at 1700 r.p.m.), involving 50-hour bench tests, made it apparent that there was little relation between Conradson Carbon Residues and carbon formation. When the engine carbon data were plotted against the 90% distilled temperature (vacuum distillation at pressure of 1 mm. of mercury), it seemed that these temperatures served as a satisfactory indication or *carbonization index* of the tendency of the oils to form combustion-chamber carbon.

A second series of tests were done with engine No. 2, a 3$\frac{1}{4}$ in. by 5 in., six-cylinder overhead poppet-valve engine and a number of oils differing in volatility, oxidation and carbon formation under severe cracking conditions.

The results obtained confirmed the inability of Conradson Carbon residue values to indicate carbon forming tendencies and the practicability of the carbonization index.

These workers adopted the principle of considering the total carbon, as formed, and not the weights of carbon on an oil-free, ash-free basis, since the heat-insulation effect of the deposit is due largely to the deposit as a whole. They also advance the view that too many opportunities for the escape of oil exist in any engine to justify the assumption of a general relation between carbon formation and oil consumption, provided normal engine conditions prevail. Data are provided in support of this.

In a discussion of the effect of varying engine conditions, results are given to show that the relationship between carbon formation by two oils varies with the engine load, e.g., over a test period of 50 hrs., at rather light or heavy loads, no great differences are apparent between oils of radically different volatilities. At light loads, combustion temperatures are so low that the rate of carbon formation is very small for both oils.

As the load increases, more rapid rates of carbon formation operate and show a difference between the oils. At higher loads, the temperatures reach a point where evaporation (and oxidation ?) prevent either oil from forming large amounts of carbon. Maximum carbon formation for the two oils studied occurred at between 50% to 60% maximum load at 1700 r.p.m., Engine No. 1, water outlet temperature and oil temperature being kept constant. With Engine No. 2, differences between both oils were apparent over most of the range of load, maximum carbon formation occurring at about 10—20% maximum load. Hence at light and heavy loads, carbon deposition is not profoundly affected by oil characteristics.

The effect of time of duration of test was also studied. The carbon formed appears to reach an equilibrium value after approximately 100 hrs. Equilibrium probably represents the thickness of deposit required to provide sufficient heat-insulation to raise the surface temperature to the point at which the oil will be evaporated rather than to remain for subjection to cracking and oxidation. An oil of high carbonization index would need a thicker insulating layer of carbon to reach this condition.

The paper indicates that no one set of arbitrarily chosen engine conditions can be counted on to give a reliable comparison of carbon formation by oils, and that at least 50 hrs. and preferably 100 hrs. should be the length of test. Details are given of the vacuum distillation apparatus used and also of a pressure regulating valve of the capillary resistance type. C. I. K.

1458. I.C. Engines of the Liquid Fuel Injection Compression Ignition Type. H. R. Ricardo and W. M. Bergius. E.P. 355,812–3, 31.8.31. Appl. 29.5.30.

The invention relates to liquid fuel injection compression ignition engines in which the gases pass to and from the combustion chamber through a common port, controlled by a pressure sealing valve, to the chamber containing the inlet and exhaust valves. By keeping this valve continuously open during the starting period, the effective compression ratio of the engine is lowered, and it can be started with a combustible vapourised charge fired by a sparking plug. Also, the inlet valve is operated so that it closes substantially later than the pressure sealing valve. Thus the throttling effect on the air charge due to its flow through two valve controlled ports is minimised.

In the second patent the charge is forced, at the end of the compression stroke, into a combustion chamber of circular cross section, in a plane at right angles to the cylinder axis, and whose diameter is smaller where it opens into the cylinder bore than the diameter of the latter. The fuel injection devices are disposed in the wall of the combustion chamber at points remote from its axis, so as to inject sprays which have a substantial component in a direction parallel to the combustion chamber axis.

The whole charge in the combustion chamber is caused to rotate about the axis of the chamber by providing at the mouth of the combustion chamber, and at the piston face opposite to it, annular projecting and recessed parts which interengage when the piston is approaching the end of its compression stroke. The charge is forced through openings, provided in the circumferential walls of these interengaging parts, inclined from the cylinder axis.

Several constructions for providing this rotary movement are detailed. J. M. F.

1459. I.C. Engines of the Liquid Fuel Injection Type (Combustion Chamber Improvements). P. Clerget. E.P. 357,519. Appl. 22.5.30. Conv. (France), 25.5.29.

A shape of combustion space to operate on the 2-str. cycle is described, in which the fuel is injected into a chamber situated to one side of the axis of the cylinder. The scavenge valve is located in this side chamber, and the exhaust valve in the part of the head opposite the chamber. This arrangement gives rise to a rotational swirl of the air. The fuel is sprayed perpendicularly to the axis of this movement, and strikes a projection which causes it to divide into two parts having gyratory movements perpendicular to the gyratory movement of the air stream. Perfect atomisation of the fuel is obtained by the interaction of these movements.

This form of combustion head can be efficiently air cooled. J. G. W.

1460. I.C. Engines of the Liquid Fuel Injection Compression Ignition Type (Combustion Chamber Improvements). H. R. Ricardo. E.P. 357,586. Appl. 25.6.30.

Comp. ign. engines having a cylindrical combustion space co-axial with and of smaller diameter than the working cylinder, in which the fuel is injected parallel to the axis of the cylinder, and at a distance from it and in which

the air is given an axial rotational swirl, suffer from the disadvantage that the air in the region of the combustion chamber walls is chilled, due to its high velocity, to such an extent that incomplete combustion takes place, giving rise to rough running and carbonisation.

The invention consists of the use of metallic liners, separated from the pressure walls by an air gap or other insulating material and covering all possible parts of the combustion chamber. The heat flow can by their means be greatly reduced.

Several methods are described for locating the liners in engines having the combustion chamber contained in the head, in the piston crown or having one combustion space common to two co-axial working cylinders. J. G. W.

1461. Producing a Petrol Gas. W. G. Harrold. E.P. 357,846, 1.10.31. Appl. 14.11.30.

A carburetting device is described in which a centrifugal fan causes the drum rotor of a meter to rotate, while volatile liquid hydrocarbons are spread over the surface of the drum. It is claimed that the constant presenting of a fresh surface to the fuel to be vapourised results in a mixture of air and vapour of constant composition even though the load be varied considerably. H. C. R.

See also Abstract No. 1449.

General.

1462. Cleaning Petroleum Storage Tanks. *A.P.I. Accident Prevention Manual No.* 1—August, 1931.

Employees engaged in the cleaning of petroleum storage tanks may suffer accidental injury as a result of explosion or fire, the presence of toxic gases, a deficiency of oxygen, the failure of a tank or its appurtenances, the misuse or failure of tank cleaning tools or equipment. The more serious accidents usually result from explosion, fire or asphyxiation. The causes of these accidents and the steps which should be taken to prevent them are discussed in detail.

The explosion limits of gasoline or crude oil vapours are given as 1%—6% by volume of vapour in air, although with butane the upper limit may be 8·5%, and for natural gas between 5—15%. For H_2S the explosive limits are 4.3%—46.0% in air. Sources of explosion are :—electric spark resulting from a discharge of accumulated static electricity on the tank or equipment surfaces, from direct lightning strokes or induced charges from nearby flashes, or from worn and defective insulation on electrical equipment. Explosions may also arise from sparkes or flames produced by tools, etc., or from the pyrophoric oxidation of iron sulphide under the roof of the tank. Atmosphere containing more than 0·2% of petroleum vapour is dangerous to breathe for long periods of time (in high concentration there is insufficient oxygen to supply the bodily needs, resulting in asphyxiation), while 0·1% of the H_2S in air may cause unconsciousness and death owing to its paralytic effect on the lungs.

Other possible causes of injury are detailed and a complete list of the steps to be taken during cleaning to avoid injury is given. The necessity for the use of gas masks until the concentration of vapours is below 0·2% (or less than ·01% of H_2S) is stressed, together with the wearing of life lines

in case of asphyxiation. Non-sparking tools should be used if the concentration of vapours is above this figure. Care should be taken when cleaning tank walls with caustics to avoid getting it into the eyes, while the hands should be smeared with mutton tallow when light petroleum solvents are to be used for cleaning. C. L. G.

1463. Petroleum in 1929. G. R. Hopkins and A. B. Coons. Min. Res. U.S. Pt. II, 421—521.

The production of crude in the U.S. in 1929 was 1,007,323,000 brls., a new high record and an increase of 12% over 1928. The production of natural gasoline was 52,271,000 brls. and of benzole 3,055,000 brls. Imports of crude and refined oils amounted to 108,710,000 brls., so that allowing for differences in stocks at the beginning and the end of the year, the indicated domestic demand for all oils was 940,083,000 brls., an increase of 9% over 1928. The total value of the crude produced in 1929 was 1,280,417,000 dollars and the average value per brl. 1·27 dollars, as compared with 1·17 dollars in 1928. Data on domestic production, imports, stocks, world production, values and the number of new wells drilled are given for the years 1918/1929 inclusive. The domestic demand for each grade of oil (*i.e.*, gasoline, kerosine, etc.) is shown in graphical form for each of the years 1918/1929. Tables are provided showing the crude production by fields or districts, and also by States, and important developments in each State during the year are commented upon. A detailed analysis of the number of wells drilled in the various States and their average daily production is given, and a list showing the average gravity of the oil produced in each State is provided.

The production of petroleum and the royalties from wells under Federal control and the rate of taxation of crude production in various States are tabulated. The quantity of crude petroleum distilled in the U.S.A. in 1929 was 987,708,000 brls., an increase of 8% over 1928. The quantities of gasoline, kerosine, gas oil, fuel oil, lubricating oil and paraffin wax produced in 1929 are also given and the figures analysed. The experimental plant of the Bureau of Mines of Colorado was the only shale oil unit operated in the U.S. owing to the low prices of petroleum products. The world production of oil shale by countries for the years 1925/1929 is given in tabular form. R. A. E.

1464. Lining the Inner Surfaces of Liquid Containers with Bituminous, Resinous, Wax-like or Similar Substances. R. Rostock, V. Baerlocker and G. Walter. E.P. 357,594, 25.9.31. Appl. 25.6.30.

The inner surfaces of liquid containers of large capacity such as fermentation vats or tanks for storing liquids such as beer, wine and so on and made of hydraulic mortars, concrete or brickwork, may be lined with bituminous, resinous or wax-like substances by applying the protective agent in the form of an emulsion. The operation is preferably carried out in two stages; a layer consisting of a stable emulsion with considerable penetrating power is first applied and when this has penetrated to a sufficient depth and has become fairly dry, is followed by a second layer of an unstable emulsion. The surface may be finished by the action of a flame which gives a shining surface. If desired, fillers may be used in the emulsion. S. F. B.

1465. Burning Liquid Fuels. F. Watson. E.P. 357,689, 1.10.31. Appl., 22.7.30.

A petrol burner is described consisting of a filter, a vaporiser, a peripherally apertured burner and a needle valve for flame control. The filter contains carborundum and shavings of nickel alloys possessing a heat co-efficient of less than 0·08, *e.g.*, Monel metal. H. C. R.

Coal and Shale.

1466. Chemistry of the Estonian Oil Shale "Kukersite." P. N. Kogerman. Monograph, Univ. of Tartu, 1931. (Reprint. Archiv für die Naturkunde Estlando, X (1). 2).

A summary of a very complete study of the chemistry of Estonian oil shale. There are three main divisions: (1) the oil shale, "kukersite"; (2) the shale oils, "kerogen"; (3) hydrogenation of the oil shale and the shale oil. The author also advances evidence for the common origin of oil-shales and petroleum.

The methods of chemical investigation of oil shales employed were:—(1) the action of reagents; (2) the action of solvents; (3) the action of heat. The action of potassium hydroxide did not produce any conclusive evidence, but fusion with sulphur gave rubber-like materials. A comparison of the results of the action of nitric acid on kukersite and the alum shale indicates that the organic material of kukersite and alum shale indicates that the organic material of kukersite may be characterised as bituminous, humic acids hardly being present at all. Chlorination indicated the possible presence of hydro-aromatic hydrocarbons in the kerogen. Kukersite is little affected by ordinary organic solvents. The action of heat revealed that there is no so-called "free bitumen" in the kukersite, and the oils obtained on distillation of the shale are decomposition products of the kerogen. Tables are given showing the yield of gases and oils obtained from kukersite at various temperatures and using various methods of distillation. Vacuum distillation did not increase the yield of oil but gave a more viscous oil, probably more unsaturated and with a lower phenol content. When the ultimate compositions of extracts and vacuum oils of kukersite and some natural asphalts were compared, a close similarity was observed, leading to the assumption that asphalt-base oil shales may be the source of some asphalts in nature.

The general characteristics and the chemical composition of the shale oil have been examined. The sp. gr. of representative samples of kerogen varies within the limits 0·990 to 1·009 at 15° C. The average ultimate composition is of the following order:—C, 81·26%; H, 10·15%; O, 7·26%; S, 1·08%, and N, 0·25%. The dry oil contains approximately:—neutral oils (chiefly hydrocarbons) 72·1%; phenols, 22·4%; carboxylic acids, 4·0%; bases, 0·2%. The boiling range depends almost entirely upon the method of retorting; the samples of kerogen investigated were obtained from a Pintsch internally heated retort.

Analysis of kerogen indicated the presence of large quantities of phenolic compounds; O–, M–, and P–, cresols were identified, the presence of 1:4:5–, 1:2:4–xylenols and higher phenols, derivatives of naphthols and anthrols assumed. Phenol ethers of the guaiacol type are also present. The acid content of kukersite oil is one of its characteristic features, and in this respect it differs widely from low temperature coal tars; the acids are mainly fatty acids. The bases isolated apparently belonged to the pyridine and quinoline series and were present only in small amounts. A number of saturated hydrocarbons were isolated from the light shale oil, the normal paraffins predominating. Part of the light oil was soluble in liquid SO_2 and probably contained diolefines since rapid autoxidation was observed, it also contained naphthenes. The higher fractions were found to have good lubricating properties. The presence of ketones was proved qualitatively in a wide range of fractions.

Hydrogenation of the kukersite oil gave products identical with the oils obtained by direct distillation of kukersite. Therefore it is suggested that the greater part of the molecules of kerogen of kukersite are built up either of concentric layers or of long spiral chains.

The composition of shale oils appears to be very similar to the composition of petroleum oils; the majority of compounds found in kukersite oil have also been isolated from well-petroleum. A common origin is therefore suggested, the well oils being formed from oil shales by mild pressure and heat. The rôle of plankton organisms in the formation of oil shale deposits, of natural asphalt, and petroleum is stressed. Those compounds of this raw material most resistant to decomposition, *viz.*, waxes and resins with decomposition products of proteins and cellulose on one side, and putrifaction products of undigested organisms on the other, it is suggested have participated. The organic matter of kukersite might be regarded as a highly polymerised "resinic substance" mixed with calcium salts of fatty acids; it is insoluble in ordinary organic solvents and only decomposed by heat and cone nitric acid. From the colloid chemical point of view the mineral is of the "irreversible coagulum" type. W. D. S.

See also Abstract No. 1433.

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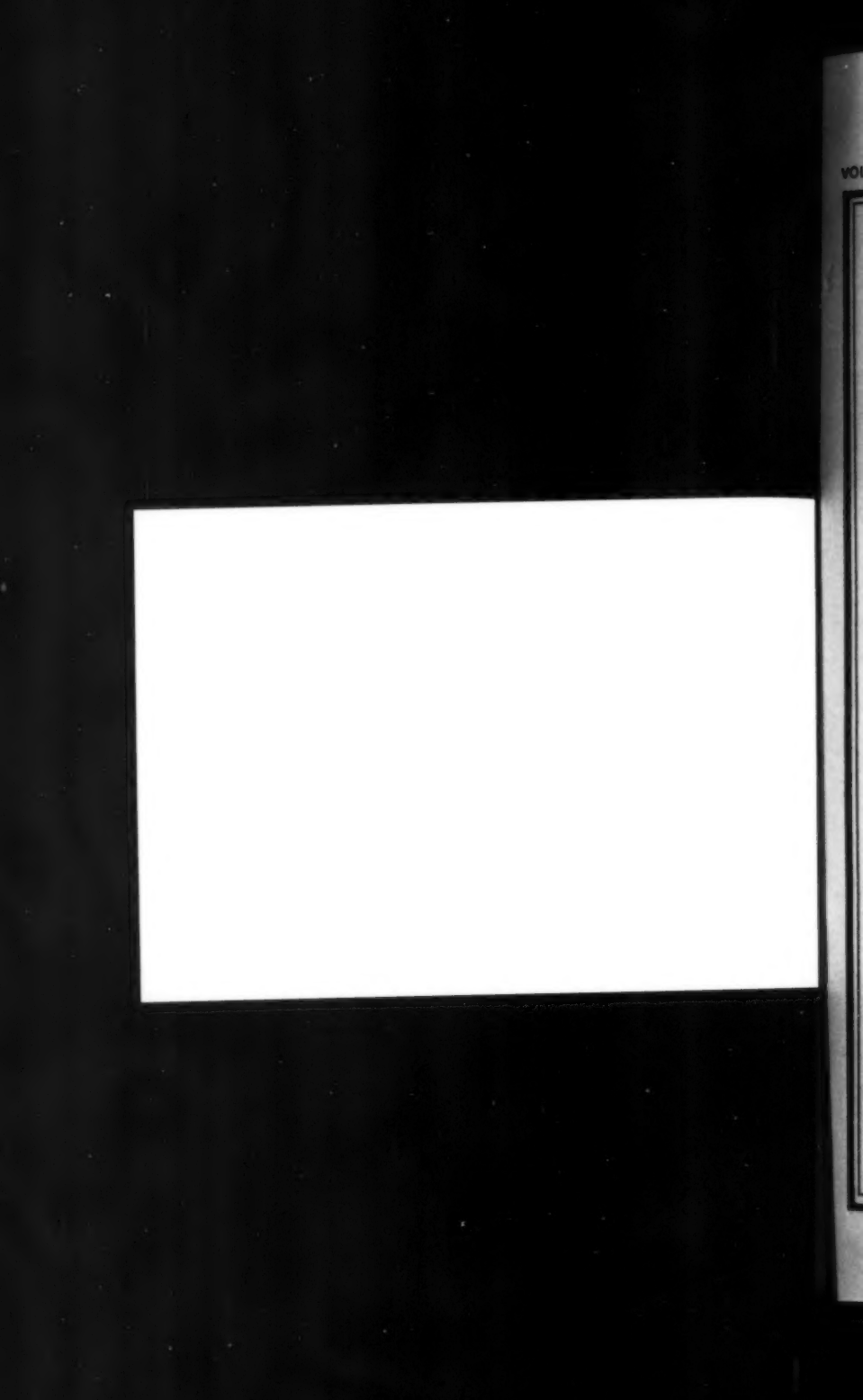

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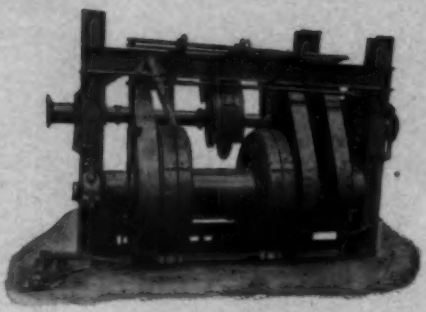

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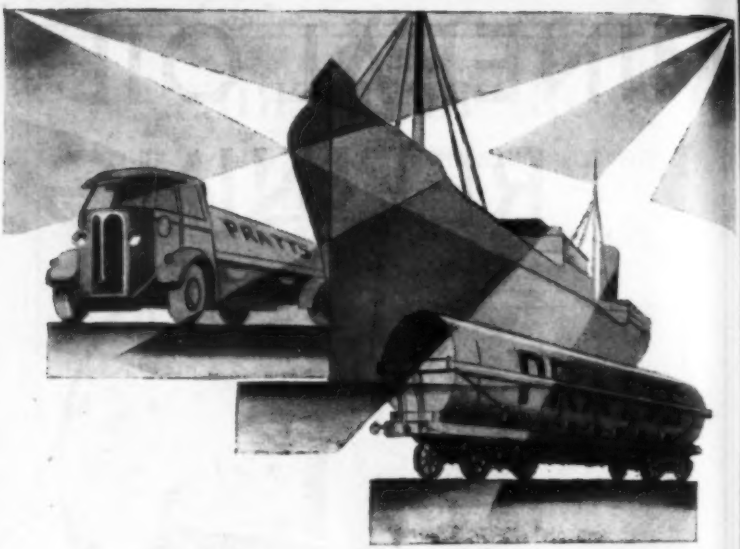

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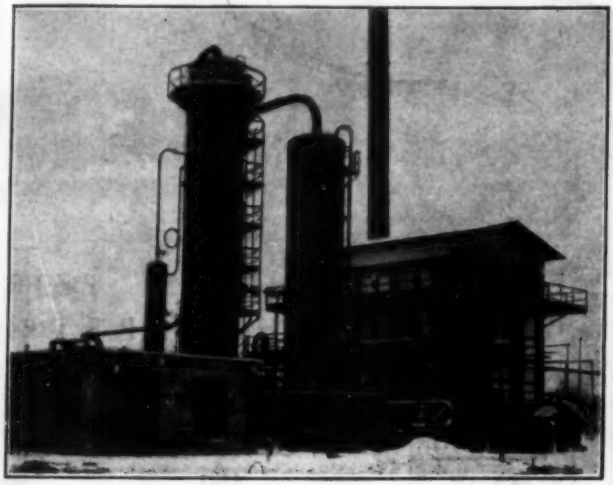

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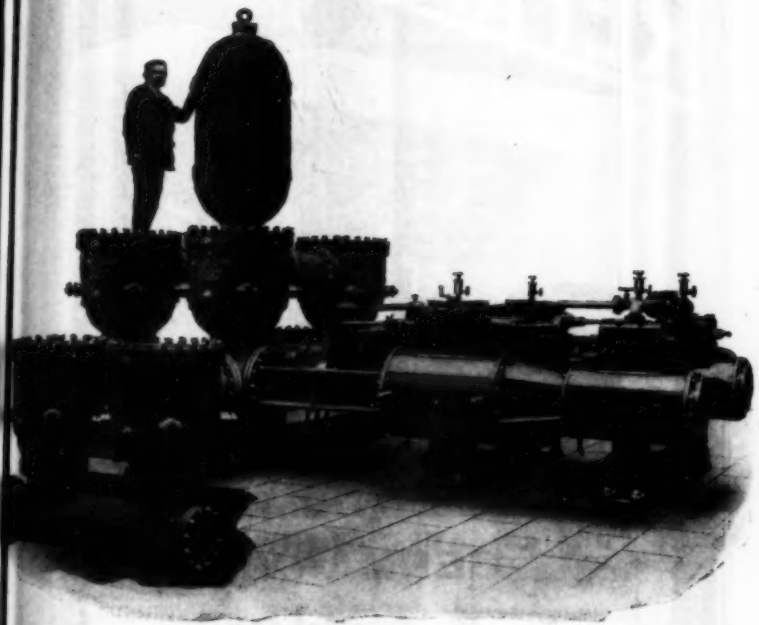

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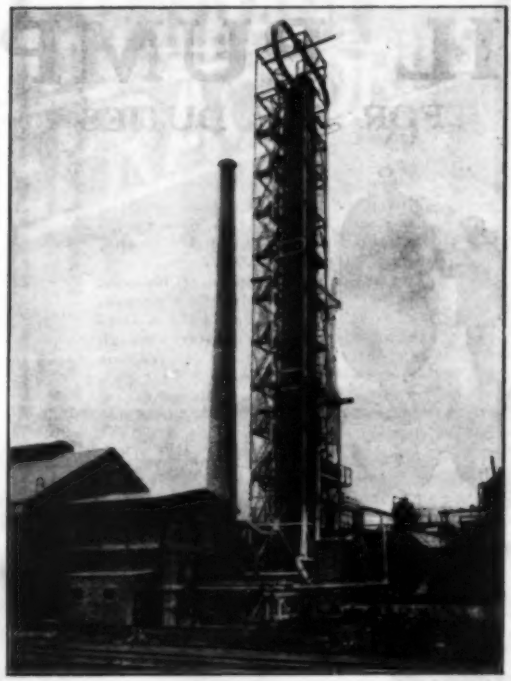

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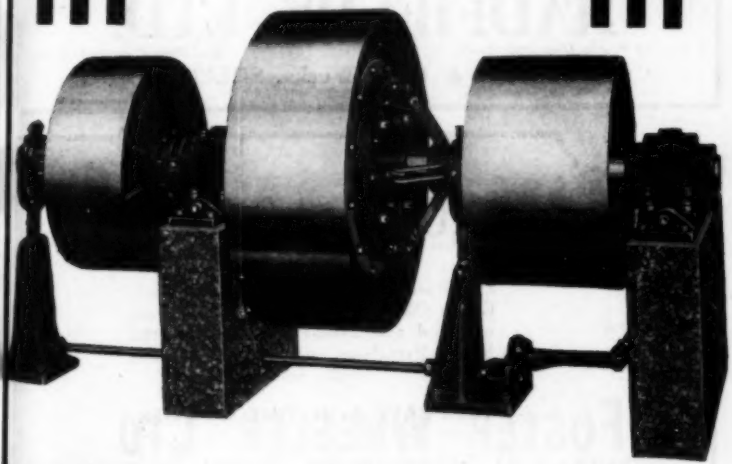

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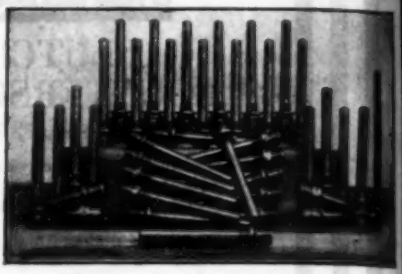

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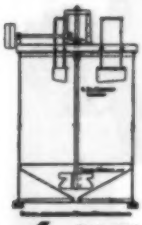

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